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# ANALYTICAL ABSTRACTS

## 1.—GENERAL ANALYTICAL CHEMISTRY

*General reviews of progress, reagents and methods of general application.*

810. **Correlation of physical constants and chemical structure. Graphical statistical methods for the identification of mineral oils, glass, silicones, fatty oils and catalysts. VI. Graphical statistical methods for studying catalytic processes. Characterisation of catalysts by parameters.** H. I. Waterman, C. Boelhouwer and J. Cornelissen (Technol. Univ., Delft, The Netherlands). *Anal. Chim. Acta*, 1958, **18** (5), 497–509 (in English).—A catalysed reaction can be represented graphically as a pseudo-ternary system of which the chosen components are the starting material, the required product and by-products. The course of the reaction is represented by a hyperbola. Examples treated are hydrogenation of linoleic esters, hydro-isomerisation of paraffin wax chlorination of benzene and hydrotreating of shale petrol. One experiment with a catalyst does not define the hyperbola, but does enable an area to be defined which includes all possible product distributions with that catalyst when one reaction variable is changed. Catalysts can thus be screened by comparing the limiting reaction courses possible with each. A second experiment with the selected catalysts enables the hyperbolas to be constructed for a more precise comparison. G. BURGER

811. **Water absorption of active aluminium oxide.** G. Wohlleben (Chem. Lab. der Fa. M. Woelm, Eschwege, Germany). *J. Chromatography*, 1958, **1** (3), 271–273 (in German).—Storage of alumina has no influence on its chromatographic behaviour, but $> 2$ hr. should be allowed after the addition of deactivating water before use. The quantity of water consumed in re-hydration must be allowed for when the Karl Fischer titration method is used to determine the activity. A. R. ROGERS

812. **Use of neutron activation analysis in analytical chemistry.** G. W. Leddicotte, W. T. Mullins, L. C. Bate, J. F. Emery, R. E. Druschel and W. A. Brooksbank, jun. (Oak Ridge Nat. Lab., Tenn.). *U.S. Atomic Energy Comm.*, Rep. AA/CONF.15/P/927, 1958, 16 pp.—The use of activation analysis at Oak Ridge Nat. Lab. is reviewed. The ORNL programme has provided much information on the method's practical use in the analysis of many different type samples. It has been used in reactor technology studies to determine U in low-grade ores, argon and xenon in reactor cooling water, and trace elements in reactor structural materials. It has also been used to determine trace elements in radio-isotope "target" materials, O in metals, S and P in metals, fine chemicals and organic materials, and As in petroleum. Other materials such as animal tissue, petroleum, grass and grain, glass, ceramics, and metals and alloys have been analysed for other trace elements. Activation analysis has also been coupled with existing sedimentation and centrifugation techniques of particle-size analysis to provide a new method of analysing particle-size distributions in samples of thorium oxide, uranium dioxide and of mixed metal oxides. So far, it has been possible to use this application to analyse samples having a particle-size distribution range of from 0·08 to 120 $\mu$. The methods of analysis used are described and typical results obtained are reported. Detailed information is presented on the use of gamma spectrometry in analysis, choice of nuclear reactions, and the techniques of the particle-size analysis method.
NUCL. SCI. ABSTR.

813. **First Conference—Analytical Chemistry in Nuclear Reactor Technology. Nov. 1957. Gatlinburg, Tenn.** *U.S. Atomic Energy Comm.*, Rep. TID–7555, 1958, 261 pp.—The following papers are included in the report. **The role of analytical chemistry in the field of atomic energy,** C. E. Larson (Nat. Carbon Co., New York), pp. 1–5. **The role of the analytical chemist in the development of power reactors,** R. B. Briggs (Oak Ridge Nat. Lab., Tenn.), pp. 6–23. **Analysis of uranium and its compounds,** C. J. Rodden (New Brunswick Area Office, A.E.C.), pp. 24–42. **Problems in the processing of raw materials for uranium,** C. F. Coleman and K. B. Brown (Oak Ridge Nat. Lab., Tenn.), pp. 43–56. **Determination of uranium in ores, leach solutions and mill products,** M. A. DeSesa (Nat. Lead Co. Inc., Winchester, Mass.), pp. 57–77. **Recent developments in the analytical chemistry of thorium and its compounds,** C. V. Banks (Ames Lab., Ames, Iowa), pp. 79–114. **Recent developments in the analytical chemistry of zirconium,** E. B. Read and F. A. Bauman (Nuclear Metals Inc., Cambridge, Mass.), pp. 116–126. **Recent developments in the analytical chemistry of titanium,** A. L. Gahler (Electro Metallurgical Co., Niagara Falls, N.Y.), pp. 127–137. **Problems in the processing of zirconium and its compounds,** W. A. DuPraw (Columbia Nat. Corp., Pensacola, Fla.), pp. 138–140. **Problems in the processing of titanium and its compounds,** W. E. Few (Cramet Inc., Chattanooga), pp. 141–157. **The analytical chemistry of beryllium,** E. S. Melick (Brush Beryllium Co., Elmore, Ohio), pp. 158–174. **Analytical control of heavy water in reactor moderator systems,** E. M. Zemyan (Du Pont de Nemours & Co., Savannah River, Augusta, Ga.), pp. 175–183. **Analysis of reactor-grade graphite,** H. G. MacPherson (Oak Ridge Nat. Lab., Tenn.), pp. 184–191. **The determination of trace elements in reactor materials by the methods of neutron activation analysis,** G. W. Leddicotte, W. T. Mullins, L. C. Bate and J. F. Emery (Oak Ridge Nat. Lab., Tenn.), pp. 192–215. **X-ray methods of analysis,** L. S. Birks (Naval Res. Lab., Washington, D.C.), pp. 216–226. **Spectrochemical analysis**

**of reactor materials,** C. Feldman (Oak Ridge Nat. Lab., Tenn.), pp. 227–238. **Solvent extraction,** J. C. White (Oak Ridge Nat. Lab., Tenn.), pp. 240–255.

814. **Sodium hexametaphosphate as a reagent in quantitative analysis.** R. Ripan, C. Stanisav, G. Marcu and M. Vancea. *Rev. Chim., Acad. R.P.R.*, 1958, **3** (1), 13–17 (in French).—(*i*) *Separation of $PbSO_4$ from $SiO_2$*—Sodium hexametaphosphate (**I**) dissolves $PbSO_4$ to form soluble complexes of the type $Na_4[Pb(PO_3)_6]$ and $Na_2[Pb_2(PO_3)_6]$, silica not being attacked. By destroying the complex by boiling with $H_2SO_4$, the Pb is re-pptd. as sulphate. The method may be used in the analysis of lead sulphide minerals. (*ii*) *Separation of $PbSO_4$ from $Au^{3+}$ and $Te^{4+}$*—In the oxidative decomposition of complex minerals containing Pb, Au and Te, the $PbSO_4$ formed may be dissolved in **I**, the Au is pptd. with oxalic acid, and the filtrate is treated with $H_2S$ in an alkaline medium to precipitate PbS and form $Na_2TeS_4$; the $Na_2TeS_4$ may be treated with $SO_2$ to precipitate Te. (*iii*) *Separation of $Pb^{2+}$ from $Bi^{3+}$*—In the pptn. of $PbSO_4$ in the presence of $Bi^{3+}$, traces of $Bi^{3+}$ may be co-pptd. By dissolving the ppt. with **I** and re-pptg., the Bi remains in soln. By boiling the filtrate with conc. $HNO_3$, Bi is pptd. as $BiPO_4$. (*iv*) *Separation of $Pb^{2+}$ from $SO_4^{2-}$*—By boiling **I** with $BaCl_2$, the barium salt is formed, which with $PbSO_4$ dissolves Pb as the soluble complex and precipitates the $SO_4^{2-}$ as $BaSO_4$. This separation may be used for the determination of S in lead minerals. (*v*) *Separation of $Fe^{3+}$ from $Cr^{3+}$*—**I** forms soluble complexes with $Fe^{3+}$ and $Cr^{3+}$, that with $Fe^{3+}$ being decomposed in the presence of aq. $NH_3$ and $NH_4Cl$ by $H_2S$, while Cr remains in soln. Dissolve the FeS on the filter with HCl (1 : 1), oxidise the Fe with hot conc. $HNO_3$, add aq. $NH_3$ and ignite the ppt. to $Fe_2O_3$. Remove excess of $H_2S$ from the Cr-containing filtrate, reduce the vol. and oxidise the Cr to $CrO_4^{2-}$ with $KMnO_4$. Destroy excess of the $KMnO_4$ by boiling, and estimate the $CrO_4^{2-}$ by addition of iodide and titration of the liberated iodine with $Na_2S_2O_3$. Representative data are given for all these separations. It is also possible to separate $Fe^{3+}$ from $Al^{3+}$. C. D. Kopkin

815. **Titrations with lead(IV) acetate.** A. Berka and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague). *Chem. Listy*, 1958, **52** (5), 926–929.—It has been found that a soln. of $Pb^{IV}$ acetate can be used as a volumetric, oxidimetric reagent for titrations not only in glacial acetic acid, but also for determinations in soln. of mineral acids in acetate buffer soln. The potentiometric titration of hydrazine and its derivatives as well as of ascorbic acid has been examined. *Procedure*—Dissolve hydrazine (2 to 10 mg), phenylhydrazine (8 to 30 mg), *p*-nitrophenylhydrazine (10 to 40 mg), semicarbazide (7 to 25 mg) or isoniazid (5 to 25 mg) in water (30 ml) and titrate with 0·05 *M* $Pb^{IV}$ acetate. For the titration of *p*-nitrophenylhydrazine a medium of 20% $H_2SO_4$ must be used and, for isoniazid, a medium of 10% $H_2SO_4$; 2 moles of the reagent react with 1 mole of the compound. For the titration of ascorbic acid (10 to 35 mg), a medium of 50% acetic acid containing an excess of K acetate (2 g) is recommended. *Preparation of the volumetric soln.*—To 1 litre of glacial acetic acid add, with stirring and in small portions, 50 g of powdered red lead; the temp. should be kept at 55° to 60°. Filter, cool and determine the factor by potentiometric titration of a known amount of quinol, the purity of which has been determined by titration with $K_2Cr_2O_7$. J. Zýka

816. **Present state of X-ray analysis.** K. I. Narbutt. *Zavod. Lab.*, 1958, **24** (5), 604–613.—The review presented covers X-ray fluorescence and absorption analysis, and micro-apparatus for X-ray spectrographic analysis. (89 references.) G. S. Smith

817. **Analytical reactions of some amidoximes. IX. *m*-Nitrobenzamidoxime.** M. Kuraš and J. Bartoň (Chem. Inst. of Pedagog. School, Olomouc, Czechoslovakia). *Chem. Listy*, 1958, **52** (5), 975–976.—*m*-Nitrobenzamidoxime (**I**) has been prepared from *m*-nitrobenzonitrile by reaction with hydroxylamine hydrochloride and $Na_2CO_3$ and its analytical properties have been investigated. A 0·1 *M* ethanolic soln. of **I** reacts with various cations, forming ppt. with $Ag^+$ (yellow ppt. in aq. $NH_3$, turning black to metallic Ag), $Hg^{2+}$ (yellow ppt. in Na acetate soln.), $Cu^{2+}$ (greenish ppt. in Na acetate soln.) or coloration with $UO_2^{2+}$ (orange - red) and $Fe^{3+}$ (reddish brown). **I** reacts also with ethanolic soln. of $Au^{3+}$ (orange ppt. in Na acetate soln., turning black), $Pd^{2+}$ (orange ppt. after boiling), and $Ni^{2+}$ (red colour after addition of 3% $H_2O_2$). The salts of **I** with $Cu^{2+}$, $Hg^{2+}$, $Pd^{2+}$ and $Ni^{2+}$ have been isolated and analysed. J. Zýka

818. **Substituted benzidines and related compounds as reagents in analytical chemistry. XIV. 3-Bromo- and 3 : 3′-dibromo-benzidine as redox indicators.** R. Belcher, A. J. Nutten and W. I. Stephen (Chem. Dept., The Univ., Birmingham, England). *J. Chem. Soc.*, 1958, 2336–2338.—3-Chloro- and 3 : 3′-dichloro-benzidine give sluggish end-points as redox indicators in the titration of $Fe^{2+}$ with $Cr_2O_7^{2-}$ or $Ce^{4+}$, but the 3-bromo- and 3 : 3′-dibromo-benzidines give sharp reversible end-points at room temp. At 60° in the titration of oxalic acid with $Ce^{4+}$ some oxidation beyond the meriquinonoid stage occurs, and the end-point is unsatisfactory unless an excess of indicator is added, which affects the titration value. Transition potentials for 3-bromobenzidine are from 892 mV in 0·5 *M* $H_2SO_4$ to 920 mV in 4 *M* $H_2SO_4$, and for 3 : 3′-dibromobenzidine 920 mV in 0·5 *M* $H_2SO_4$ to 965 mV in 8 *M* $H_2SO_4$. In HCl the yellow colour of the $FeCl_3$ interferes. The addition of $H_3PO_4$ is unnecessary in the $Fe^{2+}$ - $Cr_2O_7^{2-}$ reaction. E. J. H. Birch

819. **Preparation and analytical properties [as complexometric indicators] of NN-di(carboxymethyl)-aminomethyl derivatives of some hydroxyanthraquinones.** R. Belcher, M. A. Leonard and T. S. West (Chem. Dept., The Univ., Birmingham, England). *J. Chem. Soc.*, 1958, 2390–2393.—Complexometric indicators are prepared by a Mannich-type reaction of di- and tri-hydroxyanthraquinones with iminodiacetic acid and formaldehyde in NaOH soln. The properties of the alizarin compound, 1 : 2-dihydroxyanthraquinon-3-ylmethylamine-NN-diacetic acid (**I**), are investigated in detail. Those cations that give coloured compounds with alizarin do so with **I** and in addition **I** forms chelates with other ions. **I** is red at pH 7 to 10, mauve at pH 11·5, and violet at pH 12·5, and, if added to a soln. of an ion at a lower pH, tends to give the colour of the next pH range. At pH 4·3 good reversible end-points are obtained

with $Pb^{2+}$ and $Zn^{2+}$ at room temp., and at pH 4·0 with $Co^{2+}$ at 70° to 80° when titrating with EDTA (disodium salt) (with $Cu^{2+}$ in conc. soln. the blue colour of the Cu - EDTA complex interferes). In comparison with xylenol orange, **I** gives a poorer colour response but is preferable in more acid soln. (pH 3·8 to 4·3). Screening with Xylene cyanol FF improves the visual end-point. Small amounts of alkaline-earth metals, $Mg^{2+}$, $As^{3+}$, $Ag^{+}$, $Be^{2+}$ or $Cr^{3+}$ do not interfere, but $Al^{3+}$, $Fe^{3+}$, $Sn^{2+}$, $Sn^{4+}$, $Ce^{3+}$, $Ni^{2+}$, $Mn^{2+}$, $La^{3+}$, $Sc^{3+}$, $Th^{4+}$, $U^{4+}$ and $U^{6+}$ interfere seriously. Bivalent Cu, $Cd^{2+}$ and $Bi^{3+}$ do not affect the end-point but are titrated with the subject ion. The 0·5% indicator soln. is prepared by adding 2 drops of conc. aq. $NH_3$ to the powder, followed by 7 to 8 drops of 20% ammonium acetate soln. and diluting to vol. This soln. is stable for several months. E. J. H. Birch

820. **Analytical application of iron(III) hydroxamates. I. General reaction conditions in aqueous solution.** W. Pilz (Abt. der Farbenfabriken Bayer A.-G., Leverkusen, Germany). *Z. anal. Chem.*, 1958, **162** (2), 81–92.—A method is described for the determination of acetic acid esters based on the spot test of Feigl *et al.* (*Mikrochemie*, 1934, **15**, 9) in which, following reaction of the ester with hydroxylamine in alkaline soln. the resulting hydroxamic acid is treated with $Fe^{III}$ to form a highly coloured chelate compound, the extinction of which is measured at 490 mμ. In order to obtain uniform conditions, work was confined either to aq. soln. or to soln. containing $\geq$ 10% of water-miscible solvents. A maximum error of $\pm$ 0·3% is claimed. D. F. Phillips

821. **Preparation of metal-free acids, alkalis and buffer solutions of high purity.** H. Irving and J. J. Cox (The Inorg. Chem. Lab., South Parks Road, Oxford, England). *Analyst*, 1958, **83**, 526–528.—Concentrated metal-free soln. of HCl and $NH_3$ are best prepared by isopiestic distillation. Hydrochloric acid (500 ml) is placed in a 4 to 6-litre desiccator in which a grill of glass rods replaces the usual zinc platform and supports a polyethylene vessel containing 250 ml of conductivity water. The lid of the desiccator is not greased and the apparatus is set aside at room temp. for several days. The concn. attains 2 *N* in 2 days and 3 *N* in 4 days. With $\simeq$ 50 ml of water, 10 *N* is attained in 3 days. With 500 ml of aq. $NH_3$ and 50 ml of absorbing water, 9·5 *N* is attained in 2 days, and with 250 ml of water, 8·7 *N* in 4 days. Acid and alkali so prepared are suitable for the preparation of metal-free buffer soln. The method is not practicable with $HNO_3$ and acetic acid, but is applicable to HBr and HI and, with suitable adaptations, to the preparation of soln. of $H_2SO_3$, ammonium salts and chlorides. A. O. Jones

822. **Dithizone as an indicator for the direct EDTA titration of various metals.** A. Celse Costa (Escola Politecnica da Univ. da Bahia, Brazil). *Chemist Analyst*, 1958, **47** (2), 39–40.—Several metals were titrated in aq. media containing acetone, pyridine or ethanol. Success is claimed for the titration of Zn, Cd, Pb, Bi and Ni. Other metals tried but found to be unsatisfactory were $Fe^{II}$, $Mn^{II}$, $Sn^{II}$, $Cu^{II}$, $Hg^{II}$ and Ag.
G. S. Roberts

823. **Separation of substances with closely similar properties by displacement methods, using ion exchangers. I. General study.** B. Trémillon (Lab. de Chim. Anal., École de Phys. et de Chim., Paris). *Bull. Soc. Chim. France*, 1958, (4), 502–508.—By mathematical analysis it is shown that displacement isochrones have a bilogarithmic symmetrical form, both in frontal analysis and in development by displacement. The calculations were confirmed experimentally, but it is impossible to predict the spread of these isochrones exactly, because the height of the equivalent theoretical plate varies considerably not only with the conditions of the mechanical operation of the column, but also with the kinetics of the exchange reaction, and hence with the nature of the ions exchanged. It was found to be unnecessary to carry out highly quantitative reactions in order to obtain sharply defined separation fronts, as postulated by Spedding *et al.* (*J. Amer. Chem. Soc.*, 1955, **77**, 6125). The height of the plate can be maintained at less than a few mm.

**II. Development by displacement.** B. Trémillon. *Ibid.*, 1958, (4), 508–511.—The results of the general study have been applied to the method of development by displacement. For the separation of substances of closely similar properties, it is difficult to interpret quant. the isochrones obtained during the development stage, but when equilibrium is reached the problem of the distribution of the concn. in the band can be solved by calculation. The theoretical predictions were confirmed experimentally with $Ni^{2+}$ and $Co^{2+}$ as examples of closely related substances. J. M. Jacobs

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*See also Abstracts*—**852**, Gossypin as reagent for Be. **943**, NN-Dimethylpiperidinium iodide as reagent for Pd. **944**, 2-Mercaptobenzothiazole as reagent for Pd. **945**, 2-Mercaptobenziminazole as reagent for Pd. **967**, *p*-Bromobenzoyl *iso*thiocyanate as reagent for amines.

## 2.—INORGANIC ANALYSIS

*General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.*

824. **Multiple spots in chromatography with two competing anions.** F. Modreanu, S. Fişel and A. Carpov. *Rev. Chim., Acad. R.P.R.*, 1958, **3** (1), 147–163 (in Russian).—Ascending paper chromatography is used in the study of the effect of two competing anions from strong acids. The behaviour of Th, $UO_2^{2+}$, Al, Fe, Cr, Cu, Ni, Co, Bi, Pb, Ag, Hg, Mn, Cd, Zn (as nitrates, usually in 0·1 *N* $HNO_3$, and as chlorides, usually in 0·1 *N* HCl and sometimes as acetates), and Sb, As and Sn (as chlorides in HCl soln.) is studied. For the mobile phase, mixtures of varied composition containing trichloroacetic acid and 95% ethanol were used, both freshly prepared and also after standing for a week to give the equilibrium content of the ester. Other mobile phases used were ethyl trichloroacetate saturated with water, the same containing trichloroacetic acid, and mixtures of trichloroacetic acid with *n*-butanol, *n*-pentanol and *n*-hexanol saturated with water. Diagrams are given of the chromatograms obtained, which are developed by standard reagents. Most of the cations form tails, but Pb, Ag, Hg, Sb and Sn generally remain well grouped. The formation of double spots is not automatic even though two strong anions are competing for the cations. Increase of concn. of trichloroacetic acid in its mixtures with

ethanol causes decreased migration of the cations and leads to more compact spots. Mixtures of Pb, Hg and Ag, and Sb and As may be separated by using as mobile phase a mixture of 95% ethanol and trichloroacetic acid (25 ml to 10 g), allowed to stand for 7 days. The spots are located with ammonium sulphide. The use of longer-chain alcohols is favourable to the formation of double spots; this is particularly marked with Th; with 4- and 5-carbon alcohols, Fe and Cr form multiple spots. In general, Cd exhibits the greatest tendency to form double spots. The use of nitrates or acetates causes concentration of the spots in the upper portion of the chromatogram, because of greater conversion of the salts into the trichloroacetates and the greater mobility of the latter; when using chlorides, conversion into trichloroacetates is lessened and the formation of double spots is less pronounced. Thus the formation of double spots depends on the nature of the original anion, the nature of the cation, and the nature and composition of the mobile phase. C. D. KOPKIN

825. **Thermolysis of some transition metal chelates with 2-o-hydroxyphenylbenzoxazole.** W. W. Wendlandt (Texas Technological College, Lubbock, U.S.A.). *Anal. Chim. Acta*, 1958, **18** (6), 638–640.—The thermal decomposition of the 2-*o*-hydroxyphenylbenzoxazole chelates of Cd, $Cu^{2+}$, $Co^{2+}$ and Ni have been studied on a thermobalance, and it has been shown that these chelates possess excellent thermal properties. The first weight losses appear in the 165° to 285° range and the minimum metal oxide levels appear in the 500° to 595° range. F. L. SELFE

826. **Separations involving sulphides. V. Separation of bismuth, cadmium and indium from various other elements that form thio salts.** G. B. S. Salaria (Univ. of Allahabad, India). *Anal. Chim. Acta*, 1958, **18** (6), 520–523.—The separations are effected either by pptg. the sulphides of Bi or Cd with 2 *N* $Na_2S$ or that of In with 5 *N* ammonium sulphide while the thio salt of the accompanying metal remains in soln., or by using a sufficient concn. of ammonium sulphate or acetate and HCl to prevent pptn. of Bi, Cd or In sulphides while the thio salt is decomposed by heating. Bismuth is separated from Te, Mo, Sb and Re, respectively. Cadmium is separated from Pt, Au, Se, Re, As, Mo and Te. Indium is separated from Pt, Au, Se, Re, As, Mo, Te and Sb. All the metals are recovered quant. as their sulphides. H. N. S.

827. **Determination of aqueous solutions of hydrogen peroxide with potassium permanganate and its application to X-ray radiochemistry.** H. Izadian and P. Mergault. *Compt. Rend.*, 1958, **246** (10), 1547–1550.—The colorimetric method for determining microgram amounts of $H_2O_2$ with $KMnO_4$ ($\simeq 5 \times 10^{-4}$ *N*) can be extended to radiochemical samples (0·5 to 1 ml) containing $10^{-7}$ g of $H_2O_2$ per ml. At these concn. the error is $\pm$ 50%. The formation of $H_2O_2$ from twice-distilled $H_2O$ when irradiated for 1 to 3 hr. with X-rays, 1 cm distant in air, was followed by this method. The yield of $H_2O_2$ is independent of the material of the tube (quartz, Pyrex glass, platinum) containing the $H_2O$. Any induced coloration of the glass or quartz is also without effect. W. J. BAKER

828. **The determination of dissolved argon in water.** E. L. Shirley, C. F. Pachucki and L. K. Lolos (Knolls Atomic Power Lab., Schenectady, N.Y.). *U.S. Atomic Energy Comm.*, Rep. KAPL-1890, 1957, 18 pp.—Dissolved argon in the primary coolant water of a nuclear reactor presents a problem of accessibility to the reactor at shut-down. Because of the production of radioactive $^{41}A$ from the naturally occurring argon, it is desirable to limit the concn. of argon in the make-up water by some means of de-aeration. Two methods for the analysis of dissolved argon at low concn. are described. Both methods depend on a mechanical separation of the dissolved gas from the water and an analysis of the separated gas by a General Electric mass spectrometer. Data are shown for recoveries from standard samples which demonstrate a standard deviation of $\pm$ 0·02 p.p.m. NUCL. SCI. ABSTR.

829. **Radiometric determination of krypton-85.** R. B. Regier (Phillips Petroleum Co., Idaho Falls). *U.S. Atomic Energy Comm.*, Rep. IDO-16442, 1958, 13 pp.—Gas samples ranging in specific activity from about $1 \times 10^{-3}$ $\mu$C per ml to 10 mC per ml are analysed with only a single dilution. The apparatus and procedure are described in detail and the method of determining the absolute disintegration rate of $^{85}Kr$ is outlined. The results obtained by the radiometric determination of high-activity samples are compared with those obtained with a mass spectrometer. In the comparison of 11 such samples, the average of the radiometric to mass ratios was 1·01 $\pm$ 0·105 at the 95% confidence level. The precision of the procedure is 7·1% at the 95% confidence level. K. R. C.

830. **Spectrographic determination of lithium in its minerals.** R. Cyprès and R. Wollast (Univ., Brussels, Belgium). *Bull. Soc. Chim. Belg.*, 1958, **67** (5–6), 171–184.—From 0·2 to 10% of Li in spodumene, petiolite, lepidolite or amblygonite can be determined by fusing the sample (1 g, 100 mesh) with $Na_2CO_3$ (5 g) at 800° to 1000°, whereby a uniform solid soln. of $Li_2O$ (2 to 8%) in nepheline is formed in all instances. The cool melt is ground finely, mixed with an equal wt. of pure graphite and after arc-excitation (220 V, 8 amp.) the intensity of the Li line (3232 A) is recorded in a Hilger spectrograph and measured with the aid of a microphotometer enlarger. The plates are calibrated with the 3194-A line of Cu excited at 10 amp. for 5 sec., whilst the Li content in the sample is obtained by reference to calibration curves for the intensity ratios Li 3232 : Ag 3194 A for different synthetic mixtures. A correction is made for any dehydration, *e.g.*, of lepidolite. The error is from 4 to 10%. W. J. BAKER

831. **Determination of lithium in rocks by the method of stable isotope dilution.** A. A. Smales and R. K. Webster (A.E.R.E., Harwell, England). *Anal. Chim. Acta*, 1958, **18** (6), 587–596.—The method has been applied to the determination of Li in two standard rocks. If allowance is made for possible errors in the calibration of the tracer solutions and in the determination of the natural lithium abundance ratios, it is likely that the values obtained are correct to within at least $\pm$ 5%. Good agreement is shown by the results obtained by this method and by spectrographic analysis for china clay containing 0·1 to 0·3% of Li. The coeff. of variation for four results is usually $\pm$ 1 to 2%. The method has been used for the determination of Li in the range 1 $\mu$g to 3 mg. F. L. SELFE

832. **Determination of nitrogen in lithium.** N. I. Sax, N. Y. Chu, R. H. Miles and R. W. Miles (Nuclear Dev. Corp. of America, White Plains, N.Y.). *U.S. Atomic Energy Comm.*, Rep. NDA-38 (Del.), 1957.—The N in lithium is considered to be present as lithium nitride, and the analysis depends on the hydrolysis of the nitride to form $NH_3$ and LiOH. The $NH_3$ is distilled off with the aid of a flow of argon, collected in 5 ml of 1% $H_2SO_4$, and determined colorimetrically with Nessler reagent. Nesslerised samples are tested for opalescence by checking for $NH_3$ content at two wavelength settings of the spectrophotometer or colorimeter, far enough apart to show a displacement due to opalescence. NUCL. SCI. ABSTR.

833. **Determination of oxygen in lithium metal.** N. I. Sax and H. Steinmetz (Oak Ridge Nat. Lab., Tenn.). *U.S. Atomic Energy Comm.*, Rep. ORNL-2570, 1958, 10 pp.—The method is similar to that used to determine O in calcium (*cf.* Eberle *et al.*, *Anal. Abstr.*, 1956, **3**, 354). The apparatus is designed so that it can be sealed off from the atmosphere and the soln. is manipulated by means of excess pressure of purified A. Anhyd. methanol (usually 60 ml) is transferred, by means of gas pressure, to the reaction flask containing the washed lithium sample (0·5 to 1·0 g). A portion (usually 40 ml) of a 50% soln. of salicylic acid in pyridine is pre-titrated to an end-point with Karl Fischer reagent. The methoxide soln. is then combined with the salicylic acid and any $Li_2O$ present liberates $H_2O$, which is titrated with Karl Fischer reagent. This method is specific for O and of sufficient sensitivity to determine 10 to 100 p.p.m. of oxide. K. R. C.

834. **Determination of hydroxide in lithium hydride.** H. A. Friedman (Carbide and Carbon Chem. Co., Oak Ridge, Tenn.). *U.S. Atomic Energy Comm.*, Rep. Y-978 (Del.), 1953 (Decl. 1957), 16 pp.—For the analysis of lithium hydride, hydroxide may be determined by Karl Fischer titration of water liberated by neutralisation of the hydroxide with benzoic acid in methanol medium. Lithium oxide and lithium carbonate also react to form water, but only carbonate is corrected for because the wt. of $O^{2-}$ and $OH^-$ are very nearly the same. The major source of error is from atmospheric contamination during sample handling, but may be overcome by employing a dry argon atmosphere. Initial standardisation of the Karl Fischer reagent is with Na acetate trihydrate, and daily standardisations are made with soln. of water in methanol. Indications of precision and accuracy are implied in data from analyses of lithium hydroxide, lithium carbonate, and two sets of mixtures of lithium hydride and lithium hydroxide. NUCL. SCI. ABSTR.

835. **Analysis of lithium metal, hydride and hydroxide. V. Photometric determination of iron.** H. A. Friedman (Carbide and Carbon Chem. Co., Oak Ridge, Tenn.). *U.S. Atomic Energy Comm.*, Rep. Y-1060 (Del.), 1953 (Decl. 1957), 10 pp.—Trace quantities of Fe in lithium hydride, hydroxide and metal may be determined photometrically at a controlled pH by reduction to $Fe^{II}$ with hydroxylamine hydrochloride, and formation of the ferrous *o*-phenanthroline complex. If the concn. of Fe is very low, preliminary concentration by carrier pptn. of ferric hydroxide on zirconium hydroxide may be necessary. Lithium does not appear to interfere with the determination. On the basis of ten aliquots of an *M* LiOH soln. spiked with 1 $\mu$g of Fe per ml, the limit of error at the 95% confidence level was found to be $\pm 5{\cdot}1\%$. NUCL. SCI. ABSTR.

836. **Flame-photometric determination of sodium in uranium ores.** H. Kramer and L. J. Pinto. *U.S. Atomic Energy Comm.*, Rep. NBL-143, 1958, 17–23.—A rapid routine method was developed for application to siliceous uranium ores. Separations were not required and the results compared favourably with values obtained by standard gravimetric procedures. The lower limit of sensitivity was 0·001%. K. R. C.

837. **Application of the EDTA titration to the determination of the tetraphenylboron ion and its salts.** H. Flaschka and F. Sadek (Univ. of N. Carolina, Chapel Hill, U.S.A.). *Chemist Analyst*, 1958, **47** (2), 30–31.—A method is described in outline for the determination of $K^+$ and other ions that are pptd. quant. with the tetraphenylboron ion. It is based on the reaction of the tetraphenylboron ion with the $Hg^{II}$ - EDTA complex, whereby EDTA is released and is titrated in acetate buffer with standard zinc soln., with 1-(2-pyridylazo)-2-naphthol as indicator. G. S. ROBERTS

838. **Potassium - sodium separation on Wofatit KPS200 (16% hydrated).** F. Oehlmann and M. Heimer (Dtsch. Amt für Material und Warenprüfung, Halle). *Chem. Tech., Berlin*, 1958, **10** (5), 296–299.—Adsorption of $Na^+$ and $K^+$ on Wofatit KPS200 (16% hydrated) can be followed by selective elution of each ion with various concn. of HCl. A procedure for quant. separation of K in a 1000-fold excess of Na is described. Some typical results are reported. C. A. FINCH

839. **Paper chromatography of potassium, rubidium and caesium.** F. Modreanu, S. Fişel and A. Carpov (Acad. of Sci., Iaşi, Romania). *Nature*, 1958, **181**, 1618–1619.—Potassium, Rb, Cs and $NH_4^+$ can be separated by paper chromatography as yellow picrates. *Procedure*—Place 2 to 10 $\mu$l of soln. containing 0·1 to 0·5% of the mixed nitrates or chlorides on the narrow part of a Whatman No. 4 paper cut to a specified shape. Dry the spot at 60° and place on it 1 or 2 drops of a 1% (w/v) soln. of $NH_4^+$-free Na picrate in acetone. Dip the strip into a beaker containing nitrobenzene saturated with $H_2O$ and when the solvent has ascended $\simeq$ 15 cm remove the chromatogram and dry at 60°. The Na picrate remains at the origin. The concn. of the four elements can be determined colorimetrically after extracting the picrates from the cut bands with acetone. W. J. BAKER

840. **Determination of rubidium by the method of stable isotope dilution.** A. A. Smales and R. K. Webster (A.E.R.E., Harwell, England). *Anal. Chim. Acta*, 1958, **18** (6), 582–586.—The method has been applied to the determination of the rubidium contents of two rock samples and of sea water. It is considered that the values obtained lie within $\gtrsim \pm 5\%$ of the true value, and they agree, to within 3%, with the values obtained for these materials by neutron activation analysis. The isotope ratio of rubidium can be determined in a mass spectrometer by either a side-filament or a centre-filament technique. The former method enables rubidium and lithium determinations to be

combined. With the second method, the results for sea water suggest that it should be possible to determine amounts of Rb of the order of 1 p.p.m. in a sample as small as 1 mg. For both methods the coeff. of variation for four results is usually ± 1 to 2%. F. L. SELFE

841. **Determination of alkali-metal impurities in caesium salts by means of flame photometry.** N. S. Poluéktov and M. P. Nikonova (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1958, **24** (5), 528–531.—The mutual effects of alkali metals on their line intensities in flame photometry are studied. With the air - acetylene flame, Li and Na can be determined in salts of Cs by use of standard soln. that do not contain Cs, but the determination of K and Rb requires the use of standards containing the same concn. of Cs as the samples. To determine Li, Na, K and Rb in salts of Cs, three 0·625-g sample weights are placed in 25-ml flasks, two of which contain also portions, 1 and 2 ml, respectively, of a standard soln. containing 62·5 μg of Li, 62·5 μg of Na, 62·5 μg of K and 625 μg of Rb (all as $Cl^-$) per ml. The soln. are diluted to 25 ml and passed into an air - acetylene or illuminating-gas flame in a flame photometer. The intensities of the resonance lines Li 670·8, Na 589·0 to 589·6, K 766·5 to 769·0 and Rb 794·8 mμ are measured in all the spectra. Background corrections as shown in a table are deducted.
G. S. SMITH

842. **Complexometric determination of copper.** O. B. Budevskiĭ (Sci. Res. Inst. of Metallurgy and Useful Minerals, Sophia, Bulgaria). *Zavod. Lab.*, 1958, **24** (5), 535–537.—The best method of separating Cu from interfering elements before titration with EDTA is based on the use of iron powder, which is added to the boiling soln. containing Cu as sulphate. If aluminium is used the subsequent dissolution of the pptd. Cu in $HNO_3$ introduces $Al^{3+}$ which mask the end-point of the titration with murexide as indicator. Separation as $SCN^-$ gives low results and separation as $S^{2-}$ is unsatisfactory, unless the ppt. is ignited, since CuS dissolves with difficulty in $HNO_3$ and the pptd. S retains some Cu. For separation by means of metallic iron the acidity should be between 0·4 and 1·2 *N*, the amount of $(NH_4)_2SO_4$ should not exceed 3 g in 100 ml (otherwise Cu is incompletely pptd.), and for amounts of Cu > 200 mg in 100 ml the amount of iron added should be < 500 mg. Retardation of pptn. is caused by Sb, Bi and As, but the effect is not marked if their content is > 1% with a sample weight of 0·25 g. G. S. SMITH

843. **Spectrophotometric determination of copper as the 2-furoyltrifluoroacetone chelate.** E. W. Berg and M. C. Day (Coates Chem. Lab., Louisiana State Univ., Baton Rouge, U.S.A.). *Anal. Chim. Acta*, 1958, **18** (6), 578–581.—The $Cu^{2+}$ chelate of 2-furoyltrifluoroacetone is readily extracted into *iso*butyl methyl ketone, giving an extract that absorbs light at 660 mμ. From 1 to 10 mg of Cu may be quant. removed from < 200 ml of aq. soln. by a single extraction. Most other metal chelates are colourless, are not extracted, or do not absorb light at 660 mμ. Interfering elements are $Fe^{3+}$, $Ni^{2+}$, $Ce^{4+}$, $Pd^{2+}$ and $V^{3+}$. The method is shown to be suitable for the determination of Cu in brass, the average relative error being 1%.
F. L. SELFE

844. **Chelate formation of triammonium aurintricarboxylate [aluminon] with bivalent copper. II. Micro-determination of copper by the colorimetric method.** Anil K. Mukherji and Arun K. Dey (Univ. of Allahabad, India). *Anal. Chim. Acta*, 1958, **18** (6), 546–550.—*Procedure*—To the soln. of $Cu^{2+}$ add a 20-fold molar excess of freshly prepared aq. 0·00025 *M* aluminon, adjust the pH to 6·0 ± 0·5, dilute to standard vol. and set aside for 15 min. at 20° to 30°. Determine the optical density at 540 mμ and interpret the result with the aid of a calibration graph. The method is suitable for a concn. range of 1·7 to 200 p.p.m. of $Cu^{2+}$. Several common cations and anions interfere so it is recommended that other metals be first separated and that the $Cu^{2+}$ should be in soln. as chloride, nitrate or sulphate. H. N. S.

845. **Complexometric determination of copper in ores and enriched products.** S. Yu. Faĭnberg and L. N. Filatova (State Sci. Res. Inst. of Non-ferrous Metals). *Zavod. Lab.*, 1958, **24** (5), 534–535.—The sample (0·25 g of concentrate or 0·5 g of ore) is dissolved in $HNO_3$ - HCl mixture (3:1), the soln. is evaporated to a syrup, treated with 10 ml of a mixture of water, $H_2SO_4$ and conc. HCl (3:5:2, by vol.), and then evaporated to fuming. The cooled soln. is boiled with 40 ml of water and the insoluble matter is filtered off from the cooled soln. and washed with 2% $H_2SO_4$ soln. Copper sulphide is pptd. from the boiling soln. with 20% $Na_2S_2O_3$ soln., collected, dried and ignited, and the residue is dissolved in 3 ml of dil. $HNO_3$ (1:1), free from oxides of N. The soln. is evaporated nearly to dryness and diluted with 100 ml of water. After addition to the cooled soln. of 1 to 2 ml of 5% $NH_4F$ soln., 10 ml of a buffer soln. (100 ml of *N* acetic acid mixed with 900 ml of *N* Na acetate) and an amount of indicator (murexide and NaCl, 1:200) sufficient to give a yellow colour, titration is carried out with 0·05 *M* EDTA (disodium salt) until the colour changes to cherry-red or lilac.
G. S. SMITH

846. **Simultaneous determination of copper and iron.** K. J. Cathro and A. Walkley (Min. Utilisation Sect., C.S.I.R.O., Melbourne, Australia). *J. Polarographic Soc.*, 1958, (2), 36–40.—Boil 1 to 5 ml of sample soln. with a few drops of $HNO_3$, cool, add NaOH soln. dropwise until a permanent ppt. forms and re-dissolve in a drop of $HNO_3$. Transfer the soln. to a 50-ml flask, add 25 ml of a soln. containing 20 *M* ammonium acetate, 0·10 *M* 1:2-diamino-*cyclo*hexanetetra-acetic acid (Complexone IV), and 0·01% gelatin, and dilute to 50 ml with $H_2O$. Remove air with N and record the polarogram over the range −0·05 to −0·85 V *vs.* the S.C.E. If the amounts of Cu and Fe are comparable, the cheaper EDTA may be used with equal accuracy.
H. F. W. KIRKPATRICK

847. **Fluoroboric acid in sample preparation for the determination of copper in ferrous and non-ferrous alloys.** W. D. Nordling (Grinnell Corp., Providence, R.I., U.S.A.). *Chemist Analyst*, 1958, **47** (2), 45–47.—Procedures are described for preparing solutions of several types of alloys. Stainless steels can be dissolved in 50% fluoroboric acid; cast iron, simple steels and aluminium alloys in about 20% acid. Copper is found in the insol. residue and may be determined electrolytically after being dissolved in $HNO_3$. Some lead-based alloys will dissolve in fluoroboric acid - $H_2O_2$ (10 ml of 50% acid and 12 drops of 30% $H_2O_2$), or sodium

fluoroborate - $HNO_3$ [1 g of salt and 50 ml of $HNO_3$ (1:3)]. Copper may then be pptd. from alkaline soln. with 2-*o*-hydroxyphenylbenzoxazole, with citrate as complexing agent. G. S. ROBERTS

848. **Determination of copper in titanium, zirconium and their alloys.** D. F. Wood and R. T. Clark (Res. Dept., I.C.I. Ltd., Metals Div., Kynoch Works, Witton, Birmingham, England). *Analyst*, 1958, **83**, 509–516.—*Bis*cyclo*hexanone oxalyldihydrazone method*—The sample is dissolved in dil. HCl containing fluoroboric acid, and the soln. is oxidised with $HNO_3$ and boiled to expel $NO_2$. The liquid is diluted to a suitable vol., an aliquot is treated with ammonium citrate soln., neutralised to neutral red with 10% NaOH soln. and 1 ml is added in excess. Sodium borate soln. is added followed by bis*cyclo*hexanone oxalyldihydrazone soln. After suitable dilution of the soln., the extinction at 20° is measured at 595 m$\mu$ and referred to a calibration graph. The method is recommended for determining 0·005 to 3% of Cu. *Sodium diethyldithiocarbamate method*—The sample is dissolved in dil. $H_2SO_4$ containing fluoroboric acid, the soln. is oxidised with $HNO_3$ and boiled to expel $NO_2$. Citric acid and EDTA (disodium salt) soln. are added, and aq. $NH_3$ until the soln. is neutral to litmus paper. Excess of aq. $NH_3$ is then added and sodium diethyldithiocarbamate soln., and the liquid is extracted with $CHCl_3$. The extract is dried with $Na_2SO_4$, adjusted to a known vol. and its extinction is measured at 440 m$\mu$ and referred to a calibration graph. This method is recommended for amounts of Cu below 0·01%.

A. O. JONES

849. **Separation of lead and cadmium from copper by means of ethanolamine carbonate reagent.** . P. Ryazanov. *Sb. Nauch. Trud. Magnitogorsk. Gornometallurg. Inst.*, 1957, (13), 22–28; *Ref. Zhur., Khim.*, 1958, Abstr. No. 49,984.—For the separation of Pb from Cu a 1·5% aq. soln. of ethanolamine carbonate is added to the test soln. The Pb is pptd. as $PbCO_3$ and the Cu remains in soln. After hr. the pptd. $PbCO_3$ is filtered off and washed with the reagent soln. In order to remove the traces of adsorbed Cu, the ppt. is dissolved in hot Na acetate soln. (1 litre of 35% aq. Na acetate containing 5 ml of glacial acetic acid) and the Pb is pptd. again. The ppt. is dissolved in the acetate mixture, then pptd. as $PbCrO_4$ by $K_2Cr_2O_7$, and the ppt. is dissolved in dil. HCl, and titrated iodimetrically. The Cu in the filtrate is also determined iodimetrically. Cadmium is pptd. similarly as $CdCO_3$. After 3 to 4 hr., the ppt. is filtered off, dissolved in 5 to 20 ml of 0·3 *N* acetic acid and re-pptd. The ppt. is then dissolved in hot 2 *N* $H_2SO_4$, the soln. evaporated and the residue is ignited at 500° and weighed as CdO. The Cu in the filtrate is determined as before. W. ROUBO

850. **Determination of silver in uranium and beryllium oxides with dithizone.** A. R. Eberle. ..S. *Atomic Energy Comm.*, Rep. NBL-143, 1958, -11.—A colorimetric procedure was developed for se with the Friedeberg method (*cf. Anal. Abstr.*, 955, **2**, 2046). The dithizonates are extracted with $Cl_4$ at pH 2·0, the Ag is removed by back-extraction ith a NaCl - HCl soln., and then re-extracted with thizone. The silver dithizonate is determined ectrophotometrically. Results are tabulated.

K. R. C.

851. **Polarographic analysis of photographic films. Determination of silver iodide.** G. Russell (Ilford Ltd., Brentwood, Essex, England). *J. Polarographic Soc.*, 1958, (2), 25–27.—The method is based on the oxidation of the iodide to iodate, followed by polarography in alkaline soln. *Procedure*—The film (100 sq. cm) is hardened by immersion in 2·5% formaldehyde soln. for 5 min., washed briefly, blotted dry and fixed in 25 ml of satd. KBr soln. (2 to 3 min.). A 10-ml aliquot of the KBr soln. is pipetted into a 25-ml flask, and 2·5 ml of *N* KOH is added followed by 3 drops of Br. The flask is gently shaken for 5 min., excess of Br is removed by adding 0·5 ml of freshly prepared satd. aq. $Na_2SO_3$ soln., then 1 drop of 1% methyl red soln. is added and the soln. is made up to vol. The pptd. AgBr is centrifuged off and the clear soln. is then polarographed. The $E_{\frac{1}{2}}$ value for the iodate wave is $-$ 1·06 V *vs.* the mercury pool. The standard error is $\pm$ 1·5%.

H. F. W. KIRKPATRICK

852. **Gossypin—a colorimetric reagent for beryllium.** J. C. Bose, K. Srinivasulu and B. S. V. Raghava Rao (Dept. of Chem., J.V.D. Coll., Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1958, **162** (2), 93–95 (in English).—Gossypin, a glycoside of the flavanol gossypetin, forms a complex with Be, the limit of detection of which is 0·04 mg of BeO per litre. In the presence of EDTA (disodium salt), the colour reaction becomes almost selective although the detection limit rises to 0·1 mg of BeO per litre. Maximum absorption of both reagent and complex were observed in the regions 360 to 380 m$\mu$ and 420 m$\mu$, respectively. In the presence of EDTA at pH 7·0 a threefold excess of Al will not interfere and measurement at 450 m$\mu$ is optimum. The probable composition of the complex is 1:2 of Be to reagent.

D. F. PHILLIPS

853. **Determination of small amounts of beryllium in rocks and minerals by a fluorescence method with pentahydroxyflavone (morin).** C. N. Fedorchuk. *Trudy Inst. Mineral. Geokhim. Kristallokhim. Redk. Element., Akad. Nauk SSSR.*, 1957, (1), 178–181; *Ref. Zhur., Khim.*, 1958, Abstr. No. 49,989.—After the decomposition of the sample and removal of $SiO_2$, the soln. is electrolysed with a mercury cathode to remove Fe, Cu, Mn, Zn, etc.; Be and Al are then pptd. from the soln. by aq. $NH_3$. The ppt. is dissolved in NaOH soln. and the morin reagent is added. An aliquot of this soln. is taken and the yellow - green fluorescence due to the presence of Be is measured by a u.v. spectrophotometer. Smaller concn. of Be can be determined by this technique than by the quinalizarin method. The method has been applied to the analysis of granite, gneiss, greisen, shale, quartz, topaz, fluorspar, tourmaline, etc., some of which materials contained $<$ 0·001% of Be. W. ROUBO

854. **Quantitative determination of strontium-90 when mixed with other $\beta$-emitting isotopes.** G. Mayr (Milan Univ., Italy). *Ric. Sci.*, 1958, **28** (7), 1469–1472.—A photographic method, utilising an emulsion of suitable sensitivity and thickness, has been developed for determining a $\beta$-emitting isotope of low energy, *viz*, $^{90}$Sr, in the presence of $\beta$-emitters of high energy, *e.g.*, $^{90}$Y or $^{89}$Sr.

L. A. O'NEILL

855. **Microgram estimation of barium and uranium after paper-chromatographic separation.** I. I. M. Elbeih and M. A. Abou-Elnaga (Dept. of Chem., Univ. of Cairo, Giza, Egypt). *Chemist Analyst*, 1958, **47** (2), 35–36.—In this method developed for the determination of Ba and U in carnotite ores, the ions are separated on paper with phenazone - dioxan. Barium is leached from the spots and titrated with EDTA. The U is determined by visual comparison with standard spots after treatment with $K_4Fe(CN)_6$ soln. The max. error for Ba is 0·9 $\mu$g and for U is 1·5 $\mu$g in analyses of spots containing $\geqslant$ 100 $\mu$g of either ion. Ions that interfere with the Ba separation include $Sb^{3+}$, $Cr^{3+}$, $Na^+$, $K^+$, $Al^{3+}$, $Mg^{2+}$, $Sr^{2+}$, $Co^{2+}$, $Ni^{2+}$, $Ti^{4+}$, $Ag^+$, $Zn^{2+}$, $Be^{2+}$, $Mn^{2+}$, $Fe^{3+}$ and $Ca^{2+}$. Some metals will separate with U, but will not interfere with the colour reaction. G. S. ROBERTS

856. **Report on the determination of small amounts of radium in solution.** J. E. Harding, A. Schwebel and L. L. Stockmann (Nat. Bureau of Standards, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 311–315.—Procedure and apparatus employed in the radon method for the determination of small amounts of Ra are described. The radon, removed in a stream of dry N introduced through a fritted glass disc, is purified by being passed successively over heated Cu, ascarite, and $Mg(ClO_4)_2$. Counting of $\alpha$-particles is commenced after a period during which thoron decays and the radon attains equilibrium with Ra-A and Ra-C. The electrical circuits employed are specified in diagrams.
A. A. ELDRIDGE

857. **Determination of zinc in nickel electrolytes.** I. R. Klyachko and A. D. Vinogradova (Moscow Polygraphic Inst.). *Zavod. Lab.*, 1958, **24** (5), 540–541.—A visual colorimetric method based on the use of methyl violet for the determination of $\leqslant$ 0·0005% of Zn in standard nickel plating soln. [containing in 1 litre 250 g of $NiSO_4.7H_2O$, 20 g of NaCl and 20 g of $H_3BO_3$ together with impurities (Zn, Fe, Cu and $NO_3^-$)] is described. A mixture of 25 drops of water, 5 drops of a saturated soln. of tartaric acid, 2 drops of conc. HCl, 4 drops of 20% $NH_4SCN$ soln. and 6 drops of 0·06% methyl violet soln. is divided into three parts, one of which is to serve as a reference. The second part is mixed with 2 drops of the sample of nickel electrolyte. If its colour changes from bluish-green to violet or blue the content of Zn in the bath is $\leqslant 5 \times 10^{-4}$ %. The third part is mixed with 2 drops of the bath soln. diluted ten times in order to assess if the max. concn. allowed (0·005% of Zn) is exceeded. The actual content of Zn present can be assessed by suitable dilution of the sample and comparison of the colour with a scale of standards.
G. S. SMITH

858. **Potentiometric determination of zinc and cadmium when present together.** P. K. Agasyan (M. V. Lomonosov Moscow State Univ.). *Zavod. Lab.*, 1958, **24** (5), 532–534.—In soln. of HCl, potentiometric titration with $K_4Fe(CN)_6$ soln. of a mixture of $Zn^{2+}$ and $Cd^{2+}$ gives only one potential jump and this corresponds to the content of Zn. In soln. of acetic acid, separate jumps corresponding to Zn and Cd, respectively, are observed, but when soln. containing progressively lower contents of Zn are studied it is found that the first jump gradually disappears and that the second jump corresponds to the sum of Zn and Cd. Two titrations, one in HCl and one in acetic acid soln., serve to give the contents of both Zn and Cd. To determine Zn and Cd in a ZnS - CdS phosphor, the sample (0·2 to 0·3 g) is dissolved in HCl, the soln. is evaporated to dryness, the residue is dissolved in water (100 ml) and an aliquot (20 or 25 ml) is treated with acetic acid to give an acidity of 0·2 *M* when the soln. is diluted to 50 ml. The soln. at 60° to 70° is mixed with 1 ml of 1% $K_3Fe(CN)_6$ soln. and titrated with 0·02 *M* $K_4Fe(CN)_6$. A second aliquot is titrated in 0·2 *M* HCl. G. S. SMITH

859. **Determination of small amounts of cadmium in the presence of copper.** A. I. Preobrazhenskiĭ. *Sb. Nauch. Trud. Kazakhsk. Gornometallurg. Inst.*, 1957, (15), 303–310; *Ref. Zhur., Khim.*, 1958, Abstr. No. 49,991.—The reagent used is K ethylxanthate (**I**). To a soln. containing Cd and Cu, 25% aq. $NH_3$ (one-quarter of its vol.) is added followed by 0·15 g of **I** for every 10 ml of the soln. After a vigorous shaking the ppt. of Cu ethylxanthate is filtered off and the filtrate boiled for 2 to 3 min. The Cd is pptd. as CdS ($S^{2-}$ are formed from the decomposition of the reagent). Potassium butylxanthate can be used instead of K ethylxanthate, but a vol. of aq. $NH_3$ soln. equal to that of the original soln. must be added. The min. amount of Cd determined was of the order of $0·5 \times 10^{-3}$ mg per ml. A 300-fold excess of Cu does not cause interference. W. ROUBO

860. **Determination of cadmium as an impurity in zirconium and Zircaloy.** R. M. Burd (Westinghouse Electric Corp., Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-515, 1958, 6 pp.—Cadmium is extracted as the diethyldithiocarbamate complex with $CHCl_3$ from a HF - $H_3BO_3$ soln. at pH 1·0. The organic phase is stripped with $HNO_3$, and Cd is determined colorimetrically as the dithizonate. Potassium cyanide is used to mask interfering metals in the dithizone extraction. The method covers the determination of Cd in zirconium and Zircaloy in the range of 0·1 to 5·0 p.p.m. on a 2-g sample basis. NUCL. SCI. ABSTR.

861. **Rapid and precise micro-heterometric determination of mercury with *p*-dimethylaminobenzylidenerhodanine.** M. Bobtelsky and J. Eisenstadter (The Hebrew Univ., Jerusalem, Israel). *Anal. Chim. Acta*, 1958, **18** (6), 534–540.—Bivalent Hg ($\simeq$ 0·1 mg in 20 ml of soln.) is titrated at pH $\simeq$ 7 with 0·0025 *M* *p*-dimethylaminobenzylidenerhodanine (DBR) in acetone and ethanol, in the presence of 5 ml of *M* trisodium citrate. The end-point is detected heterometrically (*cf. Anal. Abstr.*, 1956, **3**, 12) with the use of a Fisher (Corning) green filter (525), and coincides with the formation of $Hg(DBR)_2$. Under certain other conditions the compounds $Hg_2(DBR)_3$ and Hg(DBR) are pptd. The titration is conducted successfully in the presence of 19 different common metal ions, each in great excess, and the max. error recorded is 1·5%. When $Au^{3+}$, $Pt^{6+}$, $Pd^{2+}$ or $Ag^+$ are present, a modified procedure is used. H. N. S.

862. **Determination of microgram and submicrogram amounts of boron. II. The separation of boron by distillation and the evaporation of distillates.** G. S. Spicer and J. D. H. Strickland (U.K.A.E.A., Woolwich, England). *Anal. Chim. Acta*, 1958, **18** (6), 523–533.—The method of separation of B by distillation with methanol is described in three variations to accommodate various characteristics of samples. Silica or platinum vessels are used throughout. Water is first removed from the

samples without appreciable loss of B, and the use of dehydrating agents in the subsequent formation of methyl borate is then unnecessary. The distillate is collected in at least half its vol. of water containing small amounts of NaOH, NaCl and glycerol. The resulting soln. is evaporated to dryness with a constant loss of 4% of B, for which the corresponding correction can be applied, the determination of B being carried out as in Part I (*cf. Anal. Abstr.*, 1958, **5**, 4019). Notes are given on the purification of reagents and on the avoidance of contamination from other sources. The limit of detection is $\simeq 0{\cdot}01$ p.p.m. and a precision better than 5% can be attained with amounts $> 0{\cdot}2$ p.p.m.
H. N. S.

863. **Determination of boron in aluminium - uranium fuel elements.** K. W. Puphal, J. A. Merrill, G. L. Booman and J. E. Rein (Phillips Petroleum Co., Idaho Falls). *U.S. Atomic Energy Comm.*, Rep. IDO-14418, 1957, 13 pp.—A rapid spectrophotometric method is described. The sample is dissolved in HCl and $H_2O_2$ and the B is determined without separation by the carminic acid method. At the working wavelength of 585 m$\mu$, U and Al form complexes with carminic acid which have weak absorbancies and therefore compensation is made by including U and Al in the blank. The U contents of the Experimental Test Reactor (ETR) may vary as much as $\pm$ 1·6% (absolute) or $\pm$ 10% (relative) without affecting the results of the determination. The coeff. of variation is about 2·5% for ETR-fuel containing 0·1% by weight of B.
K. R. C.

864. **Spectrophotometric determination of boron in uranium oxide with 1:1′-dianthrimide.** A. R. Eberle, M. W. Lerner and H. Kramer. *U.S. Atomic Energy Comm.*, Rep. NBL-143, 1958, 3–8.—Uranium and other cations are removed by cation exchange (*cf.* Martin and Hayes, *Anal. Chem.*, 1952, **24**, 182). The eluate is evaporated in the presence of pyridine to eliminate the loss of B and colour is developed with 1:1′-dianthrimide in conc. $H_2SO_4$. The absorbance of the standards and samples is measured against a standard containing no B as a reference at 620 m$\mu$ and a slit width of 0·03 mm. The results are tabulated and compared with those obtained by the methyl borate - curcumin procedure.
K. R. C.

865. **Determination of boric oxide.** V. R. Yankovskiĭ and L. V. Yankovskaya (Bereznikov Geological Prospecting Expedition). *Zavod. Lab.*, 1958, **24** (5), 538–540.—The value of the method of Brunisholz and Bonnet (*cf. Brit. Abstr. C*, 1952, 349), which is based on the use of a cation-exchange resin to remove interfering substances before a titration of borate in the presence of mannitol, is confirmed.
G. S. Smith

866. **Potentiometric determination of cations precipitated by 8-hydroxyquinoline.** G. Berraz. *Rev. Fac. Ing. Quím., Argentina*, 1957, **26**, 65–76.—Aluminium is pptd. with excess of 8-hydroxyquinoline and the excess determined by bromination. The end-point is determined by using identical platinum electrodes. Amounts of the order of 1 mg can be determined in 45 min. with an error of $< 1\%$.
G. F. Penny

867. **Polarographic determination of aluminium. I. General introduction. Polarography of Solochrome violet RS in the presence of aluminium.** M. Perkins and G. F. Reynolds (Min. of Supply, Chem. Inspectorate, Woolwich, England). *Anal. Chim. Acta*, 1958, **18** (6), 616–624.—A survey is made of the methods available for the determination of microgram amounts of Al, together with techniques for its separation from interfering ion species. The polarography of Al is discussed, and a study is reported of a method involving the modification, by Al, of the polarographic characteristics of Solochrome violet RS (C.I. 169). Interferences from other elements are given in detail and means for their removal are suggested.

**II. Determination of microgram amounts of aluminium in beryllium metal.** M. Perkins and G. F. Reynolds. *Ibid.*, 1958, **18** (6), 625–631.—The method involves the removal of the Al from the bulk of the beryllium and from certain other elements by extraction with 8-hydroxyquinoline into $CHCl_3$. After destruction of the oxinate and removal of Fe and other interfering elements by mercury-cathode electrolysis, Al is determined polarographically. Some results are presented and theoretical aspects are discussed.
F. L. Selfe

868. **Spectrophotometric determination of aluminium in plutonium - aluminium alloys.** J. M. Cleveland, jun., and P. D. Nance (Dow Chemical Co., Rocky Flats Plant, Denver, U.S.A.). *U.S. Atomic Energy Comm.*, Rep. RFP-53, 1958, 27 pp.—The method described involves the spectrophotometric determination of Al in Pu - Al alloys following the separation of the excess of Pu and small amounts of Fe from the Al by extraction of the cupferrate into $CHCl_3$. The concn. of Al is determined on a Beckman Model DU spectrophotometer by measuring the extinction of the Al 8-hydroxyquinolate in $CHCl_3$ at 385 m$\mu$. A correction is made for the small amount of Pu which is carried through the cupferron extraction and forms Pu 8-hydroxyquinolate along with Al. This is done by reading the extinction at 500 m$\mu$ and 650 m$\mu$ and using these values to correct the 385-m$\mu$ extinction reading.
Nucl. Sci. Abstr.

869. **Fluorimetric analysis. VII. Determination of gallium in silicate rocks with 8-hydroxyquinaldine.** Yasuharu Nishikawa (Chem. Dept., Fac. of Sci., Kyoto Univ., Sakyo-ku). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1958, **79** (2), 236–238.—Since Cu, $VO_3^-$, $MoO_4^{2-}$ and a large amount ($> 30$ mg) of Al interfere with the fluorimetric determination of Ga ($< 10$ $\mu$g) with 8-hydroxyquinaldine (**I**) (Ishibashi *et al.*, *Anal. Abstr.*, 1958, **5**, 2140), Ga is extracted with ether from 6 *N* HCl. Ferric iron is reduced to $Fe^{2+}$, prior to the addition of **I**, with hydroxylamine hydrochloride (**II**). Rock samples ($\simeq 0{\cdot}2$ g) are decomposed with HF (10 ml) and conc. $H_2SO$ (1 ml), the residue is fused with $KHSO_4$ (2 g), the cake dissolved in HCl (1 + 1, 30 ml) and the Ga extracted with ether (3 × 15 ml). After the ether is evaporated off, the residue is dissolved in *N* HCl (5 ml) and water (40 ml), treated with **II** (1 to 2 g) on a water bath for 10 min., cooled and mixed with **I** (1%, 1 ml) and ammonium acetate (20%, $\simeq 5$ ml) to give a pH of 3·9. The Ga - **I** complex is extracted with $CHCl_3$ (3 × 10 ml), diluted to 50 ml and submitted to fluorimetry.
K. Saito

870. **Spectrographic determination of gallium, indium and thallium in lead - zinc ores and their industrial by-products.** A. N. Bronshteĭn. *Sb. Nauch. Trud. Gos. Nauch.-Issled. Inst. Tsvetn. Met.*, 1958, (14), 51–57; *Ref. Zhur., Khim.*, 1958, Abstr. No. 49,994.—Spectrographic analysis has been carried out by the use of the lines Ga 4172·06,

In 4101·7 and Tl 3775·7 A and copper electrodes. Calibration standards are based on ZnO, ZnS, PbO, PbS, $PbSO_4$, CaO, MgO and $SiO_2$; In, Tl and Ga are introduced in the form of nitrates. The sensitivity depends largely on a suitable base. The mixture of a sample and ZnS standard base in the ratio 1:5 gives best sensitivity. For the stabilisation of the temp., 10 to 15% of $K_2SO_4$ is added. The line K 4044·1 A is used as the internal standard which gives a standard graph for all the base standards. The minimum determinable concn. is 0·001%. The presence of Fe causes "overlapping" of Fe 1472·13 A with the Ga line. The average error is of the order ± 7%. W. Roubo

871. **Colorimetric determination of traces of indium and gallium.** M. Z. Yampol'skiĭ. *Uch. Zap. Kurskogo Gos. Ped. Inst.*, 1957, (4), 116–127; *Ref. Zhur., Khim.*, 1958, Abstr. No. 49,993.—Azo dyes and dyes of the anthraquinone and triphenylmethane series have been studied for their application to the photometric determination of In and Ga. Reactions were carried out in ammonium acetate soln. buffered at pH 3 to 5·7. The best results were obtained with Eriochrome cyanine R (In, 2·0 μg per ml; Ga, 0·60 μg per ml), Eriochrome azurol (In, 3·5 μg per ml; Ga, 0·50 μg per ml) and aurintricarboxylic acid (In, 36·0 μg per ml; Ga, 0·60 μg per ml). All the dyestuffs used also give colour reactions with Al. W. Roubo

872. **The application of Trilon B [EDTA] for the separation of indium from copper, cadmium, lead, zinc and tin.** A. A. Rozbianskaya. *Trudy Inst. Mineral. Geokhim. Kristallokhim. Redk. Element., Akad. Nauk SSSR*, 1957, (1), 171–177; *Ref. Zhur., Khim.*, 1958, Abstr. No. 49,983.—EDTA forms extremely stable complexes with Cd, Cu, Zn, Pb and $Sn^{2+}$ in alkaline soln. In the pH range 8 to 10, $In^{3+}$ forms a complex which decomposes on boiling giving $In(OH)_3$ quant. This observation has been utilised in the separation of In from these metals. The soln. is pptd. with 1 to 2% EDTA (disodium salt) soln. neutralised with NaOH to Congo-red indicator. The $In(OH)_3$ is re-pptd. with aq. $NH_3$, and the ppt. is washed first with warm 1% aq. NaCl soln. and then with 1% aq. $NH_4NO_3$ soln. until all $Cl^-$ have been removed. A single pptn. gives ≃ 99·6% separation of other elements. When determining In in the presence of large amounts of Cu and Sn, a preliminary pptn. of the hydroxides of In and Sn is carried out with aq. $NH_3$. When determining small amounts of In, $Fe(OH)_3$ is used as a carrier. The hydroxides are dissolved in HCl and the $Fe^{3+}$ reduced with iron powder, and after 30 to 40 min. In is polarographed from − 0·4 to − 0·8 V. Small amounts of Sn do not interfere. This method has been used for the determination of In in sulphur and other ores.
W. Roubo

873. **Polarographic determination of indium in zinc-base alloys. II. Determination of indium in zinc and in zinc alloys of low aluminium content.** G. F. Reynolds and H. I. Shalgosky (Min. of Supply, Chem. Inspectorate, Woolwich, England). *Anal. Chim. Acta*, 1958, **18** (6), 607–611.—Studies of the application of an 8-hydroxyquinoline extraction and of a zinc oxide 'collection' procedure for the separation of In from zinc alloys are described and discussed. A polarographic method for the determination of In in zinc and in zinc alloys of low aluminium content is presented. The method is not suitable for mazak alloys containing about 4% of aluminium.

**III. Determination of indium in zinc and in "lead-free" zinc alloys.** G. F. Reynolds and H. I. Shalgosky. *Ibid.*, 1958, **18** (6), 612–615.—The use of a KI base electrolyte for the separation of the polarographic steps due to In and Cd is discussed, and theoretical data relating to the interference of the step due to Pb with that due to In are presented. A method is given for the polarographic determination of In in zinc alloys, applicable when the lead concn. is small compared with that of indium.
F. L. Selfe

874. **Titrations with quinol and analogous reducing agents. VIII. Potentiometric determination of thallium(III) salts.** E. Krejzová, V. Simon and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague). *Chem. Listy*, 1958, **52** (5), 936–938.—A sample of a thallium salt is oxidised with persulphate to the tervalent form and determined by reductimetric titration with a soln. of quinol. *Procedure*—To the dissolved sample containing 3 to 30 mg of Tl add 20% $H_2SO_4$ (5 ml) ($HNO_3$ does not interfere), dilute to 20 ml with $H_2O$, add ammonium persulphate (0·5 g) and boil for 15 to 20 min. (final vol. 15 ml). Cool and titrate potentiometrically with 0·01 *N* quinol. Near the end-point add the reagent dropwise and wait 3 min. after each addition. A ten-fold excess of Cu, Pb, Ag, Bi, Co, Zn, Al, Mg, Mo, W, As, phosphates, nitrates and sulphates as well as small amounts of chlorides do not interfere; $Fe^{3+}$, $Sn^{4+}$, $Sb^{5+}$, $Hg^{2+}$, $Cr_2O_7^{2-}$, $MnO_4^-$, $Br^-$ and $I^-$ must be absent; in the presence of an excess of Cd, low results were obtained.
J. Zýka

875. **Determination of sodium thiocyanate in the presence of arsenic by sulphur displacement method.** S. M. Golyand. *Trudy Gos. Nauch.-Issled. Inst. Prom. i San. Ochistke Gazov.*, 1957, (1), 96–104; *Ref. Zhur., Khim.*, 1958, Abstr. No. 50,029.—For the determination of NaSCN (60 to 120 g per litre) in the presence of As by the S displacement method, $SCN^-$ are oxidised in an acid medium by Br to BrCN which is titrated iodimetrically after removal of excess of Br by adding phenol or formic acid. W. Roubo

876. **Photometric determination of silica in fluxes used in electrode-welding.** V. F. Mal'tsev and L. P. Luk'yanenko (All-Union Sci. Res. Tube Inst.). *Zavod. Lab.*, 1958, **24** (5), 537–538.—The sample (0·1 g) of finely divided flux, which contains $CaF_2$, is fused with 4 g of $K_2CO_3$ and 25 g of borax (to prevent loss of Si) at 950° to 1000° for 20 min. in a covered platinum crucible, the melt is dissolved in 350 ml of water, and 7 ml of conc. $H_2SO_4$ and 6 ml of a clear 7% soln. of thiourea (to reduce and dissolve any $MnO_2$ that is pptd.) are added. The soln. is diluted to 500 ml and an aliquot (3 ml) is mixed with 17 ml of 0·15 *N* $H_2SO_4$ and 5 ml of 5% $(NH_4)_2MoO_4$ soln. and set aside for 3 min. Then to destroy the molybdophosphate complex 15 ml of 8 *N* $H_2SO_4$ is added, and to reduce the molybdosilicate complex 5 ml of a $CuSO_4$ soln. (5 mg of Cu per ml) (to act as a catalyst in the reduction) and 25 ml of 7% thiourea soln. are added, and the extinction of the soln. is measured after 3 min. (the colour is stable for 20 min.) with a red filter. A blank is carried out at the same time.
G. S. Smith

877. **Spectrophotometric determination of germanium with phenylfluorone after benzyl alcohol extraction.** A. Hillebrant and J. Hoste (Univ. of Ghent, Belgium). *Anal. Chim. Acta*, 1958, **18** (6),

569–574.—Add 5 ml of an 0·03% phenylfluorone soln. to 25 ml of a 0·5 *N* HCl soln. containing 0·05 to 0·5 μg of Ge per ml, and after 5 min. extract successively with 10 ml, 5 ml and 5 ml of benzyl alcohol. Collect the organic phase and dilute to 25 ml with ethanol. Measure the absorption at 505 mμ against a blank. Variables affecting colour development are discussed. Of possible interfering elements, $Ti^{4+}$ and $As^{3+}$ do not interfere in 100-fold excess; $Sn^{2+}$, $Sb^{3+}$, $Bi^{3+}$, $Mo^{6+}$ and $Fe^{3+}$ interfere. The coeff. of variation is 1·7%. F. L. Selfe

878. **Amperometric determination of tin with cupferron.** S. S. Yamamura, J. E. Rein and G. L. Booman (Phillips Petroleum Co., Idaho Falls). *U.S. Atomic Energy Comm.*, Rep. IDO-14436, 1958, 16 pp.—Tin is distilled from an $H_2SO_4$ medium as the tetrabromide by an acid mixture of HBr and HCl (1:3) at 155° to 165°. The Sn in the distillate is pptd. with aq. $NH_3$ soln., with the aid of an $Al(OH)_3$ carrier, to prevent chloride and bromide interference in the amperometric titration. The ppt. is dissolved in $H_2SO_4$, the soln. is adjusted to 3 *M* in $(NH_4)_2SO_4$ soln. and 2 *M* in $H_2SO_4$ and titrated with cupferron. By distilling at a slightly lower temp., and by removing Sb with a preliminary HCl distillation, any Sb or Bi interference in the titration can be eliminated. There is no significant difference between recovery results by this and by gravimetric methods at the 95% confidence level. The coeff. of variation for a single determination is 0·45%. K. R. C.

879. **Improvements in the micro-determination of tin in non-ferrous (and zirconium) alloys.** F. E. Huber, jun., D. L. Chase and E. J. Center (Battelle Memorial Inst., Columbus, Ohio, U.S.A.). *Chemist Analyst*, 1958, **47** (2), 41.—The method of Miller and Currie (*cf. Analyst*, 1948, **73**, 377), in which Sn is determined by reduction to $Sn^{II}$ by boiling with antimony and HCl in an inert atmosphere, followed by iodimetric titration, has been studied and improvements to the apparatus and titration procedure are suggested. The method has also been applied to the determination of Sn in zirconium alloys. G. S. Roberts

880. **Arsenate method for the iodimetric determination of titanium in the presence of the cations groups I and II.** G. B. Shakhtakhtinskiĭ and A. M. Mukímov. *Trudy Azerb. Ind. Inst.*, 1957, (17), 108–115; *Ref. Zhur., Khim.*, 1958, Abstr. No. 30,003.—A method has been worked out for an indirect iodimetric determination of Ti in the presence of the groups I and II elements by using sodium arsenate. The acid soln. is neutralised with 4 *N* $NH_3$ soln. to methyl orange. Dilute $H_2SO_4$ (1:2·5) (3 ml) is added, the soln. is warmed until the ppt. is dissolved, then diluted to ≃ 50 ml, warmed to 70° to 80°, and 50 ml of warm neutral 0·25 *N* sodium arsenate is added followed by 5 ml of 4 *N* ammonium acetate. After heating the mixture for ≃ 3 min. the ppt. is filtered off, washed with warm 2% $NH_4Cl$ soln. and dissolved in 20 to 30 ml of dil. $H_2SO_4$ (1:2·5). Benzene (20 ml) is added, followed by 10 ml of *N* KI, and after shaking the soln. the liberated iodine is titrated with 0·1 *N* $Na_2S_2O_3$. W. Roubo

8881. **Determination of zirconium in ores by means of X-ray fluorescence spectra.** N. F. Losev and A. N. Glotova (Irkutsk State Sci. Res. Inst. of Rare Metals). *Zavod. Lab.*, 1958, **24** (5), 619–621.—A silver anode (55 kV and 10 mA) and measurement of Zr $K_{\alpha_1}$ lines are used to determine Zr in concn. of < 0·6%. G. S. Smith

882. **Detection of uncombined oxygen in zirconium metal.** L. Silverman and W. Bradshaw (Atomics Internat., Canoga Pk., Calif.). *U.S. Atomic Energy Comm.*, Rep. NAA-SR-2633, 1958, 14 pp.—The method is the same as that described for the determination of O in helium (*cf. Anal. Abstr.*, 1955, **2**, 3342) with some modifications. Solid zirconium is dissolved in HF soln. (10%). The molecular O evolved is collected in a separate flask and finally determined iodimetrically. The oxygen held as $ZrO_2$ is not evolved and is not determined. From 50 to 170 μg (≡ 0·06 to 0·22% of O) can be determined by this method. The deviation is ±0·01% of O. K. R. C.

883. **Colorimetric determination of thorium and uranium by potassium iodate.** E. S. Przheval'skiĭ, A. P. Golovina and E. P. Nikolaeva. *Vestnik Moskov. Univ.*, 1958, (1), 171–175.—*Procedure for Th*—A soln. of Th (0·1 to 0·44 mg in 5 ml and 0·044 to 0·1 mg in 1 ml) in $HNO_3$ (25%) is heated on a water bath and a soln. of $KIO_3$ in $HNO_3$ [15 g of $KIO_3$, 50 ml of $HNO_3$ (sp. gr. 1·4), 100 ml of $H_2O$] is added until the pptn. of $Th(IO_3)_4$ is complete. The ppt. is washed with ethanol and ether, dissolved in $H_2SO_4$ (1:5) and shaken with a $CHCl_3$ - $KIO_3$ mixture. The extinction of the separated layer is measured in a Pulfrich photometer at 496 mμ. *Procedure for small amounts of U*—A soln. of $KIO_3$ (10%) is added to an acid soln. of $U^{IV}$ [0·1 to 0·8 mg in $H_2SO_4$ (4 to 5%)]. After 30 min. the ppt. is centrifuged off and washed with a dil. soln. of $KIO_3$, ethanol and ether, dissolved in $H_2SO_4$ (1:5) and extracted as for Th. K. R. C.

884. **Determination of thorium in the parts per million range in rocks.** H. Levine and F. S. Grimaldi (U.S. Geol. Survey, Washington 25, D.C.). *Geochim. et Cosmoch. Acta*, 1958, **14** (1–2), 93–97.—A 5-g sample is decomposed with a mixture of HF - $HNO_3$ followed by several evaporations with $HNO_3$ to decompose fluorides (when appreciable Mg is present up to 1·5 g of boric acid should be added to hasten the removal of the fluorides). Thorium and rare-earth fluorides are filtered off, ignited, and dissolved in $HNO_3$, and finally a soln. containing 3·75 ml of $HNO_3$ in a total vol. of 25 ml is obtained. After the addition and dissolution of $Al(NO_3)_3.9H_2O$ crystals (47·5 g) as a salting agent, the soln. is extracted twice with mesityl oxide (*cf. U.S. Geol. Surv., Bull.* 1006, 177). The organic layer is washed three times with $Al(NO_3)_3.9H_2O$ soln., the thorium nitrate is stripped from the organic solvent with $H_2O$ (2 × 45 ml) and evaporated, and the organic matter is destroyed with $HNO_3$ (10 ml) and $HClO_4$ (2 ml). The $HClO_4$ is removed by heating on a sand bath and, after adding $HNO_3$ (1:1) (6 ml) to dissolve the residue, the iodate separation (*cf.* Grimaldi *et al.*, *Anal. Abstr.*, 1957, **4**, 3622) and colorimetric determination of Th with thoron in *meso*tartaric acid (*cf.* Fletcher *et al.*, *Anal. Abstr.*, 1957, **4**, 3904) are carried out. R. A. Howie

885. **Thorium in iron meteorites: a preliminary investigation.** G. L. Bate, H. A. Potratz and J. R. Huizenga (Argonne Nat. Lab., Lemont, Illinois). *Geochim. et Cosmoch. Acta*, 1958, **14** (1–2), 118–125.—Samples (≃ 1 g) sawn from iron meteorites are placed in quartz vials and irradiated for three weeks

under a nominal slow neutron flux of $3 \times 10^{14}$ neutrons per sq. cm per sec. The sample is then etched in 6 *N* HCl to remove approx. 5% of its mass, to eliminate any surface contamination, and transferred to a 40-ml polyethylene tube containing 8 *N* HCl (15 ml), conc. HF (1·5 ml) and Zr (30 mg) as chloride, together with a known amount of $^{231}Pa$ tracer. The radiochemical assay is based on $^{233}Pa$, the 27·4-day daughter of the 23·3-min. $^{233}Th$ which is produced by neutron activation of $^{232}Th$. Chemical separation and purification of the Pa is made according to the procedure of Potratz and Bonner (U.S.A.E.C. Rep. LA-1721, 1954, 230) for which full details are given. This involves the pptn. of $BaZrF_6$ (co-pptg. Pa), dissolving the ppt. in $H_3BO_3$ and $HNO_3$, a second pptn. with $HIO_3$, solvent extraction into di*iso*propylcarbinol, and the extraction of Pa back from the carbinol phase into an aq. phase with $HNO_3$ and HF, which extraction is repeated once or twice according to the purity of the Pa as determined by the gamma spectrum. R. A. Howie

886. **Determination of thorium in uranium ores and feeds by solvent extraction employing thenoyltrifluoroacetone.** P. G. Laux and E. A. Brown (National Lead Co., Ohio, Cincinnati, U.S.A.). *U.S. Atomic Energy Comm.*, Rep. NLCO-742, 1958, 27 pp.—The thenoyltrifluoroacetone (**I**) extraction method has been investigated for the determination of microgram concn. of Th in uranium ores and concentrates. The tolerance limits for 18 elements commonly found in uranium ore concentrates were established. The effect of phosphate on the extraction of Th was studied. Cobalt, Cu, Sn and Bi were found to interfere seriously with the thoron method for the determination of Th after extraction with **I**. A **I** extraction method is described for the separation of Th and U from the interfering ions, preliminary acid sulphide and aq. $NH_3$ separations being used. The method is rapid, and results obtained compare favourably with those obtained with other procedures. Nucl. Sci. Abstr.

887. **Spectrographic determination of calcium in thorium and thoria using the Lundegårdh technique.** U.K. Atomic Energy Authority, Chemical Services Dept. (Operations Branch, Springfields Works, nr. Preston, Lancs.). U.K.A.E.A., Report IGO-AM/S-27, 1958, 10 pp.—Thorium (1 g) or thoria (≡ 1 g of Th) is dissolved in $HNO_3$ and HF. After the removal of an excess of acid, standard $Co(NO_3)_2$ soln. is added and the soln. is examined spectrographically, a flame supplied by acetylene and compressed air being used. The Ca 4226·7 A and Co 4121·3 A lines are compared and the result is derived from a calibration graph; a correction is necessary if the thorium contains Al. The method is suitable for contents of Ca from 50 to 1000 p.p.m.; the precision is about 13%. G. J. Hunter

888. **Determination of traces of uranium in thorium metal.** U.K. Atomic Energy Authority, Chemical Services Dept. (Operations Branch, Springfields Works, nr. Preston, Lancs.). U.K.A.E.A., Report IGO-AM/S-18, 1958, 6 pp.—The sample (4 g) is dissolved in $HNO_3$ containing HF. After evaporation of the soln. until crystallisation commences it is transferred to a cellulose column. The U is eluted from the column with a $HNO_3$ - ether mixture and determined fluorimetrically. The method is applicable to thorium with contents of U from 0·1 to 10 p.p.m. G. J. Hunter

889. **Analysis of bismuth-base alloys. III. Ternary alloys containing uranium and neodymium or praseodymium.** G. W. C. Milner and J. W. Edwards (A.E.R.E., Harwell, England). *Anal. Chim. Acta*, 1958, **18** (6), 513–519.—The alloys contain 0·2 to 5% of U and 0·2 to 5% of Nd or Pr. The Bi is first separated from a soln. of the alloy in *N* $HNO_3$ by extraction with a soln. of diethylammonium diethyldithiocarbamate in $CHCl_3$. The U is then extracted with the same reagent from the soln. buffered at pH 5·5 to 6·0 with ammonium acetate, and is determined absorptiometrically in the extract. The Nd or Pr remaining in the aq. soln. is determined by titration with EDTA (disodium salt) at pH 5·6 to 5·8, xylenol orange being used as indicator. The Bi is titrated in a separate aliquot of the sample soln., adjusted to pH ≃ 1·5, with EDTA (disodium salt) and xylenol orange as indicator. H. N. S.

890. **Spectrophotometric determination of iron in high-purity bismuth.** J. H. High and P. J. Placito (Hawker Siddeley Nuclear Power Co. Ltd., Langley, nr. Slough, Bucks., England). *Analyst*, 1958, **83**, 522–525.—The sample (10 g) is dissolved in conc. $HNO_3$ and the soln. is boiled, then cooled and adjusted to a known vol. An aliquot (≡ 0·7 to 1·5 g of sample) is evaporated just to dryness and again after addition of 10 ml of constant-boiling HCl. The residue is boiled with constant-boiling HCl, then cooled and adjusted to 25 ml with this acid. The extinction is measured at 390 mμ against a blank soln. treated similarly. A suitable standard iron soln. (≡ 50 μg of Fe per 25 ml) is included in each batch of samples. Beer's law is obeyed up to 500 μg of Fe per 25 ml of final soln. Recovery expt. gave satisfactory results. Interference by other elements in the amounts found in high-purity bismuth is negligible. A. O. Jones

891. **Determination of vanadium as an impurity in zirconium and Zircaloy.** H. R. Wilson, D. L. Smith and G. W. Goward (Westinghouse Electric Corp., Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-191, 1957, 4 pp.—Vanadium is oxidised with $KClO_3$ in conc. $H_2SO_4$ to a yellow compound, probably a polyvanadate, and is determined by spectrophotometric measurement of the intensity of the colour at 410 mμ. The method covers the determination of V as an impurity in zirconium and Zircaloy in the range 5 to 100 p.p.m. Interference by impurity and alloying Fe, Ni and Cr is eliminated by measurement of the absorbance of the samples against corresponding samples in which the vanadium colour is not developed. Nucl. Sci. Abstr.

892. **The determination of tantalum in niobium.** M. L. Theodore (Westinghouse Electric Corp., Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-542, 1958, 7 pp.—The metal is dissolved in HF and $HNO_3$; HCl is added and the Ta is extracted with *iso*butyl methyl ketone. The Ta is removed from the ketone layer by pptn. with aq. $NH_3$. The ppt. is dissolved in HF - HCl, re-extracted from a HCl medium and converted into the hydroxide; this is fused with $K_2S_2O_7$ and the Ta determined spectrophotometrically at 355 mμ using the tantalum - pyrogallol system. Nucl. Sci. Abstr.

893. **Photometric determination of tantalum in titanium and its alloys by means of arsenazo.** E. I. Nikitina. *Zhur. Anal. Khim.*, 1958, **13** (1).

72–78.—*Procedure*—Dissolve 0·1 g of titanium alloy in 20 ml of $H_2SO_4$ (1 : 1), oxidise with 25% $(NH_4)_2S_2O_8$ until the soln. becomes orange, then heat until colourless. If the soln. is not clear add more $(NH_4)_2S_2O_8$ until an orange colour is developed, then heat until $SO_3$ fumes are evolved and solution is complete. To the cold colourless soln. add 10 ml of 20% tartaric acid soln. and make up to 100 ml with water. To a 10-ml aliquot add 20 ml of water and 5 ml of 0·05% arsenazo soln. followed by $K_2CO_3$ soln. (10%) until any free $H_2SO_4$ is neutralised (to Congo red paper). Add 20 ml of HCl (1 : 1), heat to boiling, cool and dilute to 100 ml with water. Measure the extinction of an aliquot in a 50-mm cell with an orange filter and compare with a calibration graph. Carry out a blank determination on the reagents. The tantalum - arsenazo complex is stable for long periods and the method enables up to 15% of Ta in titanium alloys to be determined without a preliminary separation of Ti.

W. Roubo

894. **Sulphuric acid for use in lead - acid batteries. (Limits for impurities.)** British Standards Institution (2 Park St., London). B.S. 3031 : 1958, 16 pp. —Analytical methods are described for the determination of residue on ignition; chloride; sulphur dioxide; combined ammonia; nitrogen (other than combined ammonia); iron; copper; manganese; arsenic.

N. E.

895. **Colorimetric determination of sulphate ions by means of coloured complexes of zirconium and thorium.** A. K. Babko and L. V. Markova (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1958, **24** (5), 524–528.—Methods based on the interaction of $SO_4^{2-}$ with complexes of Zr and Th with alizarin and the complex of Zr with Acid Chrome blue K are described. They are applied to the determination of S in metals after combustion in O and absorption of the $SO_2$ and $SO_3$ that are formed in 0·01 *N* NaOH. Thus contents of S of $\simeq$ 0·001% in tin, $\simeq$ 0·01% in steel and alloys of Ni with Mo or W, and $\simeq$ 0·09% in ferrotungsten can be determined.

G. S. Smith

896. **Quantitative precipitation studies in aqueous - non-aqueous media. Estimation of barium sulphate.** T. Chandra Gupta and A. K. Bhattacharya (Chem. Dept., Agra Univ., India). *Z. anal. Chem.*, 1958, **161** (5), 321–323 (in English).—When the concn. of $H_2SO_4$ in the final dilution is $< 0{\cdot}064$ *N* the results are low in aq. soln., but with concn. of 0·064 *N* $H_2SO_4$ and 30% (v/v) ethanol or 20% (v/v) acetone quant. pptn. is obtained.

E. G. Cummins

897. **Determination of selenium in glass by a radioactivation method.** J. L. Putman and W. H. Taylor. *J. Soc. Glass Tech.*, 1958, **42**, T84–T86.—For the determination of Se in glass, after its addition as a decoloriser, two 13-g samples of the glass, one with and one without the Se, are exposed to a flux of $1{\cdot}2 \times 10^{12}$ neutrons per sq. cm per sec. for 3 days. The samples are aged for 3 weeks to allow the short-lived activities to decay to a low level and the activity of the $^{75}Se$ is determined by a differential scintillation counter method (NaI plus Tl crystal). A calibration is obtained from a similarly irradiated range of soln. of $SeO_2$ in $H_2O$ (order of 10 p.p.m.). An accuracy of 5% is achieved.

J. A. Sugden

898. **Polarographic determination of chromium in ores.** J. Průša and E. Skokan (Res. Inst. of Ores, Prague). *Chem. Listy*, 1958, **52** (5), 915–917.—Chromates in soln. of NaOH, NaCl and (phenylimino)-diacetic acid (**I**) yield a distinct polarographic wave, suitable for the determination of Cr in the presence of all elements present in chromium ores. *Procedure*—Mix the sample (0·2 to 2 g) with $Na_2O_2$ (5 to 7 g), place the mixture on 3 to 5 g of $Na_2O_2$ and fuse in a crucible, at first with gentle heating and then more strongly for 5 min. Cool and add the melted sample in the crucible to a 400-ml beaker containing 100 to 150 ml of $H_2O$. Dissolve the melt by heating, remove the crucible, then to the soln. add 30% $H_2O_2$ soln. (10 ml) and heat on a sand bath to remove $Na_2O_2$ and O. Transfer the soln. to a 250-ml flask and dilute to vol. with $H_2O$. Mix and filter through a filter-paper into a dry beaker. Reject the first portion of the filtrate and then measure 25 ml into a 100-ml flask, neutralise with HCl to phenolphthalein, add conc. HCl (10 ml), then *M* **I** (10 ml). Neutralise the soln. with 10 *M* NaOH, and add 1 ml in excess and gelatin (0·5%) (1 ml). Dilute to vol. with $H_2O$, mix, remove O with a stream of N and register the polarographic wave from $-$ 0·4 V. Evaluate the content of Cr by the use of the method of standard addition.

J. Zýka

899. **Determination of molybdenum and tungsten as impurities in niobium.** D. V. Reed, H. R. Wilson and G. W. Goward (Westinghouse Electric Corp., Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-620, 1958, 7 pp.—A method is presented for the determination of Mo in the range 50 to 1000 p.p.m. and W in the range 25 to 550 p.p.m. in niobium, on the basis of an aliquot representing 0·2 g of niobium sample. Toluene-3 : 4-dithiol forms coloured complexes in acid soln. with Mo and W, which are extracted with *iso*amyl acetate.

Nucl. Sci. Abstr.

900. **Contribution to the EDTA titration of molybdenum and tungsten.** J. Kinnunen and B. Wennerstrand (Outokumpu Oy, Metalworks, Pori, Finland). *Chemist Analyst*, 1958, **47** (2), 38.—Molybdenum and tungsten pptd. as calcium salts may be determined by EDTA titration, with xylenol orange as indicator. The visual end-point is poor.

G. S. Roberts

901. **Photometric method for the determination of tungsten in low-grade mine ore and mineral-dressing products.** J. B. Pollock (Geological Survey of Uganda, P.O. Box No. 9, Entebbe, Uganda). *Analyst*, 1958, **83**, 516–522.—The sample (10 g) after evaporation with HF and $H_2SO_4$ is ignited at red heat and fused with $Na_2CO_3$. The vol. of the aq. soln. of the melt is adjusted to 100 ml. A 25-ml aliquot is adjusted to pH 1·65, 25 ml of rhodamine B soln. (0·2 g per litre) is added and, after exactly 1 hr., a 1% soln. of acacia gum (5 ml) is added, the liquid is adjusted to 100 ml and set aside for 3 hr. The extinction is measured against a blank soln. at 600 m$\mu$ and referred to a calibration graph, prepared from soln. of sodium aluminate containing aliquot portions of a standard sodium tungstate soln. The blank soln., treated simultaneously with the sample, is a vol. of a standard soln. of sodium aluminate equiv. in Al content to the sample (if this is unknown, 17% is assumed) adjusted to 25 ml with 4% $Na_2CO_3$ soln. The pH value in the determination and in the calibration is critical and a procedure for its adjustment is

described. With the exception of Al, which is compensated for by the addition of sodium aluminate, elements that might interfere are absent from the low-grade material to which the method is applicable. A. O. Jones

902. **Extraction - direct spectrophotometric method specific for large amounts of uranium.** W. J. Maeck, G. L. Booman, M. C. Elliott and J. E. Rein (Phillips Petroleum Co., Idaho Falls, Idaho). *U.S. Atomic Energy Comm.*, Rep. IDO-14438, 1958, 14 pp.—An extraction - spectrophotometric procedure was developed that was specific for the determination of large quantities of U in the presence of molar concn. of diverse ions. The method is based on the quant. extraction of tetrapropylammonium uranyl trinitrate with *iso*butyl methyl ketone from an acid-deficient aluminium nitrate salting soln., followed by a direct absorbance reading of the organic phase at 452 mμ. The molar absorptivity at this wavelength is 31·4. The coeff. of variation is 0·71% at the 10-mg level. The method is simple and rapid. The uniqueness of the spectrum is useful as a qual. indication of U and as a means for readily observing interferences.
Nucl. Sci. Abstr.

903. **Spectrochemical determination of uranium-235.** W. G. Jolley (Hanford Atomic Products Op., Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-55666, 1958, 9 pp.—A method was developed for determining $^{235}U$ in concn. of 0·70 to 1·5% in enriched fuel elements. There was some evidence to show that this method would be suitable for enrichment levels of up to several per cent. The uranium sample was converted into $U_3O_8$ and it was found possible to dispense with the ZnO buffer. K. R. C.

904. **Extraction - spectrophotometric method specific for microgram amounts of uranium.** G. L. Booman, W. J. Maeck, M. C. Elliott and J. E. Rein (Phillips Petroleum Co., Idaho Falls, Idaho). *U.S. Atomic Energy Comm.*, Rep. IDO-14437, 1958, 15 pp.—An extraction - spectrophotometric method has been developed that is specific for the determination of microgram quantities of U in the presence of large concn. of diverse ions. Tetrapropylammonium uranyl trinitrate is extracted into *iso*butyl methyl ketone from an acid-deficient aluminium nitrate salting soln., followed by a single citric - tartaric - EDTA acid scrub. Colour development is made *in situ* by the addition of dibenzoylmethane in an ethanol - pyridine mixture. The molar absorptivity at 415 mμ is 16,500. The coeff. of variation is 0·71% at the 25-μg level. The lower limit of detection is 0·5 μg.
Nucl. Sci. Abstr.

905. **An extraction, controlled potential coulometric method specific for uranium(VI).** G. L. Booman and W. B. Holbrook (Phillips Petroleum Co., Atomic Energy Div., Idaho Falls, Idaho). *U.S. Atomic Energy Comm.*, Rep. IDO-14435, 1958, 21 pp.—A method, specific for U, is based on *iso*butyl methyl ketone separation of tetrapropylammonium uranyl trinitrate from acid-deficient $Al(NO_3)_3$ soln. followed by direct coulometric reduction of $U^{VI}$ at a mercury electrode. Perchloric acid volatilises Ru and Tc which are the only known interfering species extracted with U. The U is determined by controlled potential reduction of the citrate complex after a pre-reduction step to eliminate easily reducible material. The complete procedure requires ≃ 2·5 hr. Results on samples from soln. of freshly dissolved Al - U fuel elements having ≃ 20% burn-up show excellent precision and no bias as compared to the isotope-dilution thermal mass-spectrometric method.
Nucl. Sci. Abstr.

906. **Colorimetric micro-determination of uranium by means of an activated reaction.** G. Almássy and A. Kiss (Chem. Dept. of the Exp. Atomic Reactor, Central Physical Res. Inst., Budapest). *Magyar Kém. Foly.*, 1958, **64** (5), 170–173.—In weak acid soln. $Fe^{3+}$ oxidise $U^{IV}$ to $U^{VI}$ if the reaction is activated by means of 2:2′-dipyridyl (**I**), which forms a complex with the $Fe^{2+}$ obtained in the reaction. *Procedure*—Dilute a soln. containing 1 mg of U to 5 ml; acidify with 50% $H_2SO_4$ (2 ml) and, after mixing, pass the soln. through a bismuth reductor. Wash the beaker and the column with water (2 × 3 ml) and then pass sufficient water through the column to give a total vol. of collected liquid of 25 ml. To an aliquot (2·5 ml) add 0·5 ml of $FeCl_3$ soln. [12 g of $FeCl_3.6H_2O$ dissolved in conc. HCl (10 ml) and water (to 100 ml)] and bromophenol blue (1 drop, containing 0·05 g in 100 ml of ethanol). Adjust to pH 4·7 with Na acetate soln. (15%), add **I** (0·5 ml of a 0·56% aq. soln.) and dilute to 25 ml with water. Immediately measure the extinction in a Pulfrich photometer with a 5-cm cell and an S50 filter. The concn. is determined from a calibration curve. The sensitivity is ≃ 0·2 μg of U per ml. Cations that are reduced on the reductor and oxidised by $Fe^{3+}$ interfere; the U must then be extracted. The mixture of nitrates is dissolved in 0·1 *M* $HNO_3$ (25 ml) which is 6 *M* with respect to $NH_4NO_3$. To the cold soln. add tributyl phosphate (**II**) reagent (containing 24 vol. of **II** and 76 vol. of benzene, sp. gr. 0·87). Shake the mixture for 3 min., run off the lower layer and twice re-extract the upper one for 3 min. with 0·2 *M* ammonium carbonate (25 + 5 ml). Evaporate the ammonium carbonate soln. to dryness and dissolve the residue in a mixture of ethanol (2 ml), water (3 ml) and $H_2SO_4$ (1:1) (2 ml). Pass the soln. through the reductor and proceed as already described. A. G. Peto

907. **The isotopic analysis of uranium by radioactivity and spectrographic measurements.** N. E. Gordon, jun., R. A. Brightsen, H. D. Cook and C. R. Wilson (Westinghouse Electric Corp., Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-81 (Rev.), 1952, 38 pp.—An "accountability" procedure for uranium of high enrichment is outlined as a substitute for high-resolution mass-spectrographic determinations. Uranium-234 is determined by α-counting, $^{238}U$ by emission spectrography, impurities by grating spectrograph, and $^{235}U$ content is calculated by difference. Nucl. Sci. Abstr.

908. **Measurement of the isotopic enrichment of uranium by a direct comparison mass-spectrometer method.** V. L. Warren and L. A. Smith (Oak Ridge Gaseous Diffusion Plant, Tenn.). *U.S. Atomic Energy Comm.*, Rep. K-1369, 1958, 18 pp.—A direct comparison mass-spectrometer method of measuring the separation of uranium isotopes is described. Data are given to show that, for separation factors < 1·06, the accuracy and precision are equivalent to those of the commonly used hexafluoride method. The technique is useful for other separation factor ranges and is particularly adaptable to automatic instruments, which should give even better results.
Nucl. Sci. Abstr.

909. **Determination of uranium as an impurity in zirconium and Zircaloy.** D. L. Smith, H. R. Wilson and G. W. Goward (Westinghouse Electric Corp., Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-431 (Rev. 1), 1958, 5 pp.—A method for determination of uranium in Zircaloy and zirconium in the range 0·25 to 20 p.p.m. on a 1-g sample is presented. The samples are dissolved in HF and $HNO_3$, saturated with $Al(NO_3)_3$ and extracted with diethyl ether. An aliquot is fused with a mixture of NaF and LiF and the fluorescence measured. (See also *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-431, 1957.) NUCL. SCI. ABSTR.

910. **Determination of uranium, thorium and thorium-230 in sea silt.** L. A. Kuz'mina (V. I. Vernadsky Inst. Geochem. and Anal. Chem., Acad. Sci., USSR, Moscow). *Zhur. Anal. Khim.*, 1958, **13** (1), 100–106.—A procedure is described for the preparation of a soln. practically free from impurities. Thorium is determined colorimetrically after reaction with thoron. The U is determined in the soln. after the determination of the Th by acidifying with HCl and adding 20 mg of $FeCl_3$ dissolved in water. After warming, aq. $NH_3$ is added and the ppt. is filtered off, ashed and weighed, and its $\beta$-activity measured with a counter with a mica end-window. The ppt. is then transferred to an agate mortar and ground in acetone. The suspension is evaporated on a weighed disc and its weight determined. The $\alpha$-activity is then measured, from which the $^{230}Th$ is calculated. W. ROUBO

911. **Volumetric determination of uranium oxide in thorium sludge.** W. A. Peavy. *U.S. Atomic Energy Comm.*, Rep. NBL-143, 1958, 55–59.—The routine volumetric $K_2Cr_2O_7$ procedure is used but, to eliminate titration difficulties due to the high $ThO_2$ content of the sludge, the bulk of the Th is separated from the U, after dissolving the sample, with tributyl phosphate - *iso*butyl methyl ketone. The organic layer is then stripped of the U with hot water. The determination is completed by the usual method. K. R. C.

912. **Spectrographic determination of cadmium in uranium.** U.K. Atomic Energy Authority, Chemical Services Dept. (Operations Branch, Springfields, nr. Preston, Lancs.). U.K.A.E.A. Report IGO-AM/S-45, 1958, 22 pp.—Uranium (5 g) is dissolved in $HNO_3$ and, after the removal of an excess of acid, addition of citrate and adjustment of the pH to the end-point of phenolphthalein, the Cd is extracted as the dithizonate into $CCl_4$. The extract is passed through an alumina column, on the top of which the Cd is retained. The portion of the alumina containing the Cd is mixed with a carrier containing As, which is used as an internal standard, and spectrographed by using a carrier-distillation technique. The result is derived from a comparison of the intensities of the Cd 2288·0 A and As 2288·1 A lines and a calibration graph prepared from the examination of standards. The method is suitable for the analysis of uranium with contents of Cd between 0·015 and 0·15 p.p.m.; the coefficient of variation is about 10%. G. J. HUNTER

913. **Spectrographic determination of rare earths in uranium metal.** U.K. Atomic Energy Authority, Chemical Services Dept. (Operations Branch, Springfields, nr. Preston, Lancs.). U.K.A.E.A. Report IGO-AM/S-126, 1958, 27 pp.—The sample (500 g) is converted into uranyl nitrate, which is passed in an ether - $HNO_3$ mixture through a cellulose column on which the rare-earth metals are retained. After ignition of the cellulose and treatment of the residue with HF and $HClO_4$, the rare earths are twice pptd. as oxalates, Ca oxalate being used as a carrier, and once as hydroxides, a standard amount of $Fe(OH)_3$ being used as a carrier. This concentrate is mixed with four times its weight of $(NH_4)_2SO_4$ and examined on a Jarrell - Ash 21-ft. grating spectrograph set to the range 3450 to 4700 A; copper electrodes are used. Full details are given for the preparation of standard mixtures and the selection of comparison lines to enable the following limits of detection to be reached—Eu, 0·001 p.p.m.; Sm, Dy, Er, Tm and Lu, 0·003 p.p.m.; Gd and Nd, 0·005 p.p.m. G. J. HUNTER

914. **Determination of iron in uranium by the carrier-distillation method using cobalt as internal standard.** J. B. Ramsay (Los Alamos Scientific Lab., N. Mex.). *U.S. Atomic Energy Comm.*, Rep. LA-2171, 1957, 30 pp.—The spectrographic carrier-distillation method used to determine impurities in uranium has been modified to include Co as an internal standard for the determination of Fe. The precision and bias have been evaluated and compared with the original method by a study of synthetic samples ranging in Fe content from 25 to 400 p.p.m. The use of 250 p.p.m. of Co as internal standard gave a coeff. of variation of $\pm$ 9% and a bias of +6% compared with $\pm$14% and +44%, respectively, in the absence of Co. In the presence of 250 p.p.m. of Co the results for Mn were $\simeq$ 40% lower than in the absence of Co. NUCL. SCI. ABSTR.

915. **Determination of iron in ammonium diuranate and uranic liquors.** U.K. Atomic Energy Authority, Chemical Services Dept. (Operations Branch, Springfields Works, nr. Preston, Lancs.). U.K.A.E.A. Report IGO-AM/S-29, 1958, 5 pp.—Ammonium diuranate is dissolved in 8 *M* $HNO_3$, the soln. is evaporated to dryness and the residue is dissolved in water and a few drops of 8 *M* $HNO_3$; an aliquot of this soln. or of a uranic liquor containing 1 g of U is used. After the addition of sulphamic acid and ammonium carbonate to suppress interference from nitrite and U, respectively, thioglycollic acid and ammonium citrate are added and the extinction of the soln. is measured; Ilford 605 filters are used and the content of Fe is derived from a calibration graph. The method is applicable to samples containing up to 300 p.p.m. of Fe. G. J. HUNTER

916. **Gravimetric determination of uranium with the use of cupferron for its separation.** S. V. Elinson and V. A. Oleznyuk. *Zhur. Anal. Khim.*, 1958, **13** (1), 95–99.—The method, which was developed for application to ores and concentrates containing > 5% of U, is based on the separation of impurities with cupferron and pptn. of $U^{IV}$ with the same reagent after reduction with $Na_2S_2O_4$. *Procedure*—Decompose a 0·2 to 1·0-g sample in a conical flask with $HNO_3$, add 10 ml of $H_2SO_4$ (1:1) and heat until $SO_3$ fumes are evolved. Treat the residue with water so that the concn. of free $H_2SO_4$ is 10% by vol. Warm the soln. until the salts are dissolved, then cool and add dropwise 0·1 *N* $KMnO_4$ until the soln. is pink. Precipitate impurities such as Fe, V and Ti by adding 6% aq. cupferron soln. at 18° until an excess of reagent is present. Filter off the

ppt. and wash with 10% $H_2SO_4$. Dilute the filtrate to 200 ml with 5% $H_2SO_4$ and add 2 g of $Na_2S_2O_4$. Set aside for 20 min. and precipitate uranium with cupferron as described above. After 15 to 20 min. filter off the ppt., wash with a 4% soln. of $H_2SO_4$ containing 0·15% of cupferron and ignite at 1000° to 1050°. Sulphide-group elements may be removed by previous pptn. The mean square error is ± 0·3% (relative) for 50% of U and ± 1·2% for 5 to 10% of U. W. ROUBO

917. **Spectrographic analysis of uranium by the carrier-distillation method.** U.K. Atomic Energy Authority, Chemical Services Dept. (Operations Branch, Springfields, nr. Preston, Lancs.). U.K.A.E.A. Report IGO-AM/S-117, 1958, 24 pp.—Uranium oxide prepared by direct ignition of U is mixed with AgCl or NaCl and spectrographed; a graphite cup and a pointed electrode are used. Suitable standard lines are given for the determination of Au, Be, Bi, Co, Cr, Cu, Ga, Ge, Mn, Ni, Pb, Sn, Tl, V, Pd, Ba, K and Li. The limits of detection vary from 0·05 to 30 p.p.m., and the precision (= 3 × coefficient of variation) is about 50%. The preparations of standards and of spectrographically pure AgCl and $U_3O_8$ are described in detail. G. J. HUNTER

918. **Determination of chloride in uranium tetrafluoride.** U.K. Atomic Energy Authority, Chemical Services Dept. (Operations Branch, Springfields, nr. Preston, Lancs.). U.K.A.E.A. Report SCS-M-171, 4 pp.—The sample (10 g) is treated with $Al(NO_3)_3$ soln. and $HNO_3$ in a distillation apparatus (illustrated). The $Cl^-$ are distilled off and determined by adding $AgNO_3$ to the distillate, which is then compared visually with standards. The method is suitable for material with contents of $Cl^-$ from 0 to 10 p.p.m. G. J. HUNTER

919. **Determination of bromine in uranium hexafluoride.** U.K. Atomic Energy Authority, Chemical Services Dept. (Operations Branch, Springfields, nr. Preston, Lancs.). U.K.A.E.A. Report IGO-AM/S-13, 1958, 9 pp.—The sample is hydrolysed in a silver vessel and, after the addition of sulphurous acid, AgBr is pptd. and carried down on AgCl. The ppt. is treated with KOH soln. and $H_2O_2$ and after evaporation the soln. is buffered to pH 6·35; the $Br^-$ are oxidised to $BrO_3^-$ with NaOCl, excess of which is destroyed with Na formate. The soln. is then added to a mixture of KBr, ammonium molybdate, rosaniline and dil. $H_2SO_4$, and the extinction of the soln. is measured after the addition of ethanol, *tert.*-butyl alcohol and dil. $H_2SO_4$. Ilford 606 filters are used and the content of Br of the sample is derived from a calibration graph. G. J. HUNTER

920. **Measurement of impurity effects in the mass-spectrometer isotopic analysis of uranium hexafluoride.** J. C. Horton (Oak Ridge Gaseous Diffusion Plant, Tenn.). *U.S. Atomic Energy Comm.*, Rep. K-1365, 1958, 10 pp.—Tests to determine the extent of the isotopic analysis errors resulting from ion beam distortion by gaseous impurities in $UF_6$ samples were run on several mass spectrometers. Mixtures of $UF_6$ with 5 other gases were compared with pure $UF_6$ of identical isotopic composition, noting the bias of the measured ratios of mole ratios from unity. Nitrogen caused a greater bias than the other gases tested; 10% of N can cause a bias > 1% in the ratio of pure to impure sample mole ratios. NUCL. SCI. ABSTR.

921. **Detection of plutonium in wounds.** W. C. Roesch and J. W. Baum (General Electric Co., Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. A/CONF.15/P/756, 1958, 5 pp.—A problem of long standing at plants handling Pu is its detection in wounds from which the α-particles cannot penetrate. About 4% of the disintegrations of Pu are followed by emission of X-rays of about 17 keV. Thin NaI crystals are very efficient detectors of these rays (0·25 mm gives 95%) and have low backgrounds. Three cases of Pu in wounds have been studied. Depositions of 0·0014 to 0·02 μC were found. After excision or strenuous decontamination, it was shown that < 0·00004 μC was left. NUCL. SCI. ABSTR.

922. **Sensitive analytical method for the determination of very low level plutonium in humans.** L. C. Schwendiman and J. W. Healy (General Electric Co., Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. A/CONF.15/P/759, 1958, 9 pp.—A method was developed for determining extremely low levels of Pu in urine, and involves sample evaporation, ignition, dissolution of salts, co-pptn. with lanthanum fluoride, and chelation with thenoyltrifluoroacetone to isolate the Pu. An electrodeposition method subsequently separates and deposits the Pu uniformly on a small known area of a disc. Alpha-track film is used to record the emission of α-particles over a period long enough to give requisite sensitivity. After emulsion development, the tracks are counted under a microscope. A detection limit of the order of 0·03 disintegrations per min. per sample ($2 \times 10^{-3}$ g of Pu) is achieved. This detection limit permits recognition of exposure at a level of 0·005 of the maximum permissible body burden, thus ensuring timely detection of incipient deposition. NUCL. SCI. ABSTR.

923. **Spectrographic analysis of plutonium by the thenoyltrifluoroacetone extraction method.** H. H. Van Tuyl (General Electric Co., Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-28530, 1953 (Decl. 1958), 16 pp.—Impurities are separated from plutonium soln. by means of thenoyltrifluoroacetone (TTA) extractions. Plutonium in HCl soln. is reduced to $Pu^{III}$ with hydroxylamine and some impurities are extracted with hexone (*iso*butyl methyl ketone) or a soln. of TTA in hexone. The org. phase is removed and the Pu in the aq. phase is oxidised to $Pu^{IV}$ with $HNO_3$. The Pu is then extracted into TTA in hexone and most of the impurities remain in the aq. phase. The org. phase of the $Pu^{III}$ extraction and the aq. phase of the $Pu^{IV}$ extraction are then analysed by the copper spark spectrographic method. Investigations of several variables that affect the extraction are described. Results are compared with those by the carrier distillation and cupferron extraction methods. The distribution of over 60 elements in these extractions is listed. NUCL. SCI. ABSTR.

924. **Analysis for plutonium by controlled potential coulometry.** F. A. Scott and R. M. Peekema (General Electric Co., Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. A/CONF.15/P/914, 1958, 11 pp.—The method was developed for the determination of the Pu content of reactor fuels. It is superior to conventional procedures because it is independent of the plutonium isotopic content, has greater precision and sensitivity, and can be used for plant feeds and in-process soln. as well as the plutonium product. The Pu content of soln.

has been determined over a wide concn. range with a precision of ± 0·1% to ± 1% on samples containing from 2 to 0·05 mg, respectively. Either mercury or platinum electrodes are used, and the method is simple with a minimum of sample pre-treatment, and no chemical adjustment of valence state is necessary. In the course of the work, the polarographic behaviour of $Pu^{IV}$ was studied in detail in many media. Interferences have been studied, particularly of reactor fuel alloying elements and fission products, and tolerable levels of common interfering ions are defined. The method has been applied to the determination of the Pu content of metal pieces with a precision of ± 0·1% at the 95% confidence level, and also to the direct determination of the Pu content of reactor fuel processing soln. NUCL. SCI. ABSTR.

925. **Polarovoltric method for halogen ions. Determination in dilute solution.** W. Walisch and M. R. F. Ashworth (Univ. of the Saar, Saarbrücken, Germany). *Anal. Chim. Acta*, 1958, **18** (6), 632–637.—The method involves the measurement and recording of the difference between an adjustable reference voltage and the polarisation voltage set up between two platinum micro-electrodes in a solution. When the concentration of the ion or ions causing polarisation is changed by titration of the solution, the polarisation voltage changes correspondingly. The application of this method to halogen ion determination with $AgNO_3$ solution is described and results are quoted. It is stated that analysis of highly dilute halide solutions (*e.g.*, $10^{-4}$ *M*) is possible with an accuracy of about ± 0·2%. F. L. SELFE

926. **Potentiometric determination of chloride ions with mercuric nitrate.** Shih-Fu Tzou and Huan-Tsiang Tiao. *Acta Chim. Sinica*, 1957, **23** (4), 241–245.—A silver - silver chloride electrode is used as the indicating electrode while the reference electrode is silver immersed in 0·1 *N* $AgNO_3$ soln. The addition of ethanol (74% of total vol.) is advocated. SCI. ABSTR. CHINA

927. **Rapid automatic determination of microgram quantities of chloride ion.** J. H. Cannon (Food and Drug Admin., U.S. Dept. of Health, Education and Welfare, St. Louis, Mo., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 428–431.—The potentiometric determination of $Cl^-$ (down to 10 μg of Cl) in the presence of $H_2SO_4$ can be accomplished with a precision of ± 0·2 to 0·3 μg. The electrode system and stirrer, burette, pre-amplifier and potentiometer system, and the burette flow-control mechanism are specified and illustrated. A. A. ELDRIDGE

928. **Data on the volumetric determination of bromide ions.** L. Szekeres (Dept. of Gen. Chem., Univ. Agric. Sci., Budapest). *Magyar Kém. Foly.*, 1958, **64** (5), 163–165.—In a medium of $H_2SO_4$, $Br^-$ are oxidised to Br by $BrO_3^-$; the Br is then converted into $BrO^-$, which are reduced with Na formate, and the excess of $BrO_3^-$ is determined iodimetrically. *Procedure*—Mix an ≃ 0·02 *N* to 0·1 *N* KBr soln. (10 ml) with 0·1 *N* $KBrO_3$ (15 ml) and 2 *N* $H_2SO_4$ (25 ml). After 15 min., neutralise the soln. with 5 *N* NaOH (10 ml) then make it alkaline with $NaHCO_3$ (3 g). Add 0·2 *N* Na formate (10 ml) and warm the mixture on the water bath for 15 min. To the cold soln. add 2% KI soln. (20 ml) and conc. HCl (15 ml). Determine the excess of $BrO_3^-$ with 0·05 *N* to 0·1 *N* $Na_2S_2O_3$. Carry out a blank determination. Iodide ions interfere, but large amounts of $Cl^-$ do not. A. G. PETO

929. **Use of ascorbic acid in amperometric titrations. II. Determination of iodine, hypochlorites and iodates.** Z. A. Gallaĭ, V. G. Tiptsova and V. M. Peshkova. *Vestnik Moskov. Univ.*, 1958, (1), 209–213.—Ascorbic acid is quant. oxidised by iodine to dehydroascorbic acid and can be used for the titration of small amounts of iodine and also for iodimetric titrations of copper, active chlorine, chromates, permanganates and other strong oxidising agents. Sodium hypochlorite is reduced by KI and the iodine liberated is titrated amperometrically against ascorbic acid. A direct amperometric titration against ascorbic acid is also made. In both cases a phosphate buffer medium of pH 7·3 is used. Active chlorine in chloramine B is determined in an acid medium (*N* $H_2SO_4$) by the same methods. The results of collateral experiments are in good agreement. Lead and silver are determined by back-titration of excess of $KIO_3$ against ascorbic acid. Good recovery is shown. K. R. C.

930. **Amperometric determination of iodide and chloride.** M. M. Nijland and P. J. H. Eggels (Pharm. Lab., Rijksuniv., Utrecht, Holland). *Chem. Weekbl.*, 1958, **54**(22), 289–291.—Iodide and chloride are titrated separately with $AgNO_3$ soln. in one operation. A rotating silver-coated platinum wire acts as a micro-electrode and is short-circuited with a S.C.E., with which no extra external voltage is needed. The method is applicable to the determination of 2 to 8 μg of iodide per ml and of chloride 0·25 to 20 times the iodide content. The method is suitable for the titration of 20 ml of soln. P. RENTENAAR

931. **The sampling of manganese ore.** British Standards Institution (2 Park St., London). B.S. 3035:1958, 12 pp.—The recommendations are intended for the sampling of ore in freight wagons immediately before it is loaded into the ship or immediately after discharge at the port of arrival. They are applicable to all manganese ores. N. E.

932. **Titrations with quinol and analogous reducing agents. VII. Determination of the higher oxides of manganese and lead.** E. Krejzová, V. Simon and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague). *Chem. Listy*, 1958, **52** (5), 976–978.—The method described is rapid and simple and impurities in the sample (Fe and Cu) need not be masked. Standard quinol soln. is added to the sample, the excess of which is determined by cerimetric titration. The total amount of metal can be determined complexometrically after reduction with hydroxylamine. *Procedure*—To the finely powdered sample (about 20 mg of $PbO_2$, $MnO_2$ or $Mn_2O_3$) add 10 to 20 ml of 0·1 *N* quinol and 2 *N* $H_2SO_4$ (10 ml) or acetic acid (5%) (10 ml) (for $PbO_2$), and dissolve by shaking for 5 to 10 min. Add ferroin as indicator and titrate with 0·1 *N* $Ce(SO_4)_2$ till blue. To determine total metal, add to the sample (about 50 mg) hydroxylamine hydrochloride soln., 10 ml of 0·1 *M* EDTA (disodium salt), buffer soln. (pH 10) (aq. $NH_3$ - $NH_4Cl$) (3 to 5 ml) and Eriochrome black T as indicator, and titrate with 0·1 *M* $MgSO_4$ till violet - red. J. ZÝKA

933. **Spectrophotometric studies on ferrous complexes of quinoline- and pyridine-carboxylic acids. I. Ferrous quinaldate system.** Bireswar Banerjee and Priyadaranjan Ray (Indian Assoc. for the

Cultivation of Science, Calcutta). *J. Indian Chem. Soc.*, 1958, **35** (5), 297–303.—The colour of the sparingly sol. ferrous complex with quinaldic acid has its max. intensity at pH 5·8 to 7·0, and the wavelength for max. absorption is 500 mμ. The intensity of the coloured soln. increases in the presence of an excess of KCN and to some extent with pyridine, both of which make the complex readily soluble. The colour system obeys Beer's law even in the presence of KCN when the max. absorption is at 515 mμ. The composition of the coloured system is given by $Fe(C_{10}H_6O_2N)_2$ with an instability constant of $3{\cdot}63 \times 10^{-6}$. I. Jones

934. **Polarographic determination of trace elements in cast iron.** R. C. Rooney (Brit. Cast Iron Res. Assoc., Alvechurch, Birmingham). *J. Polarographic Soc.*, 1958, (2), 21–24.—Results of an examination of published methods for minor constituents with a cathode ray polarograph are discussed. Direct methods are subject to much interference and some separations are essential. Work on various separation processes is described.
H. F. W. Kirkpatrick

935. **Rapid determination of phosphorus in carbon and foundry steels by colorimetry and absorptiometry.** R. de la Cierva (Factoria de Villaverde de Manufacturas Metálicas Madrileñas, S.A.). *Inst. Hierro y Acero*, 1958, **11** (56), 85–90.—The method is a modification of that of Hague and Bright (*J. Res. Nat. Bur. Stand.*, 1941, **26**, 405). *Procedure*—Treat the sample (0·1 g) with 5 ml of acid oxidising mixture (conc. $HNO_3$ - 60% $HClO_4$ - $H_2O$) (1:3:1). Heat until white fumes of $HClO_4$ are evolved, then add $Na_2SO_3$ soln. (5%) and stir to dissolve the residue. Boil for 1 to 2 min., add 2 ml of hydrazine sulphate soln. (1·5 g per ml) followed by 5 ml of molybdic acid reagent [$(NH_4)_6Mo_7O_{24}.4H_2O$ (10 g), conc. $H_2SO_4$ (350 ml) and $H_2O$ (150 ml)]. Heat the soln. on a steam bath for 3 min. then make up to 50 ml. Measure the extinction with a Spekker H–760 absorptiometer and an Ilford 608 filter. The probable error is < 0·001% of P on samples low in P and < 0·01% P on samples high in P. Cobalt, Ni, Cr, V and Si do not interfere. Arsenic interferes but is normally not present in amounts sufficient to cause appreciable interference. The time required for a determination is 7 to 12 min. G. H. Foxley

936. **Determination of the oxygen content of steel by the Gotta method.** Collective Report of the Chemical Committee of the Vereins Deutscher Eisenhüttenleute. *Arch. Eisenhüttenw.*, 1958, **29** (6), 351–352.—The Gotta method (*cf. Ibid.*, 1943–44, **17**, 53) was recommended by earlier workers on account of its simplicity and ease of application, but the investigators found that its accuracy was less than that of the vacuum fusion process. The average values obtained varied considerably with variations of the O content, probably because of incomplete reduction of oxides. Analysis indicated that 94% of the oxides consisted of silicic acid. Since the reaction of Al in fluid steel with flint-glass inclusions is comparatively slow, it is unlikely that heating up to temperatures of 1300°, such as were used in the tests, completed the reduction of the oxide. It was therefore suggested that the small content of O in some instances was possibly due to incomplete reduction of the silicic acid. The reduction of the oxides, a function of heating times, could not be completed when prolonged heating was required as this would have resulted in an uncontrollable increase of O. With unalloyed steel, in spite of low results in the range of higher values and of occasional faulty analyses, the usefulness and reliability of the Gotta method was established. L. M. R.

937. **Determination of manganese in steels containing chromium and vanadium.** I. M. Issa, M. Hamdy and A. El Hadidy (Fac. of Sci., Cairo Univ., Egypt). *Chemist Analyst*, 1958, **47** (2), 44.—Dissolve 1 to 5 g of sample and oxidise by the bismuthate method. Adjust the acidity of an aliquot containing < 0·01 g of Mn and < 0·038 g of Cr to 0·5 *N* $H_2SO_4$ and add NaF soln. (2%) to give a concn. of 1·5%. Titrate as quickly as possible with $HgNO_3$ soln. (0·1 *N* in 3% $HNO_3$) to a visual end-point. Addition of a little $H_3PO_4$ will diminish the colour due to dissolved iron.
G. S. Roberts

938. **Group-separation method for gamma spectrometry of complex radionuclide mixtures—with special application to stainless steel and its corrosion products.** K. Samsahl (Nuclear Chem. Lab., AB Atomenergi, Stockholm, Sweden). *Acta Chem. Scand.*, 1958, **12** (6), 1292–1296.—A rapid and accurate anion-exchange method has been devised for separating alkali and alkaline-earth elements from aq. soln. of radionuclide mixtures that are to be analysed by gamma spectrometry. Short columns are coupled in series and saturated with chloride, citrate and hydroxide ions. The method has been adopted as a routine procedure for the analysis of stainless steel, its corrosion products and other elements forming impurities in the water from a water-cooled nuclear reactor. Short columns of Dowex–2 resin saturated with chloride, citrate and hydroxide ions retain, respectively, (*a*) molybdates and tungstates, (*b*) $Fe^{3+}$, $Ni^{2+}$ and $Co^{2+}$, and (*c*) $Mn^{2+}$ and $Cr^{3+}$, and the columns are then subjected to gamma spectrometry. With cobalt-containing steel it is necessary to elute the citrate resin, extract any iron from the eluate with ether and re-adsorb the cobalt on an ion exchanger for comparative measurements so that interference between $^{60}_{27}Co$ and $^{59}_{26}Fe$ in the gamma-spectrometric measurements is avoided. Corrosion products in heavy water may also be determined by the method with the addition of a Dowex-50 column in the H form after the hydroxide resin for controlling the adsorption efficiency of the other columns. By using various other forms of the resin, the method may have much wider applicability. S. M. Marsh

939. **Spectrophotometric determination of cobalt with *iso*nitroso[oximino]dimedone.** W. Van Den Bossche and J. Hoste (Univ. of Ghent, Belgium). *Anal. Chim. Acta*, 1958, **18** (6), 564–568.—The cobalt - oximinodimedone complex has a mol. extinction coeff. of 19,680 at 374 mμ and provides a means of determining Co in the concn. range 0·5 to 3 μg per ml. *Procedure*—Adjust the test soln. to between pH 4 and 7 with a phosphate buffer and extract 25 ml successively with one 10-ml and two 5-ml portions of a 0·025% soln. of oximinodimedone in *iso*amyl alcohol. Dilute the extract to 25 ml with ethanol and determine the extinction at 374 mμ in a 1-cm quartz cell. Interpret the result by reference to a calibration graph. Several common ions have little influence on the results, but $Fe^{3+}$, $Cu^{2+}$, $Cr^{3+}$ and $Ni^{2+}$ must be absent.
H. N. S.

940. **New method for the gravimetric determination of cobalt in the presence of nickel and other elements.** T. I. Pirtea, E. Antonescu and P. Bukshan (Lab. of Inorg. and Anal. Chem., Chem. Fac., C. I. Parhon Univ., Bucharest). *Rev. Chim., Acad. R.P.R.*, 1958, **3** (1), 139–146 (in Russian).—Cobalt may be separated from Ni, Zn, Mn, Cu, Pb, Cd, Hg, Ca, Sr and Ba and determined by pptn. as $[Co(NH_3)_6][Co(NO_2)_6]$. To a neutral or slightly acid soln. containing 0·008 to 0·04 g of Co (5 to 20 ml) add $NaNO_2$ (0·5 or 0·6 g) and conc. acetic acid (0·3 or 0·4 ml). Set aside for 5 min. for complete oxidation of $Co^{2+}$ to $Co^{3+}$, then add $NaNO_2$ in excess ($\simeq$ 0·7 g) to form $Na_3[Co(NO_2)_6]$ and stir vigorously to remove oxides of N. When evolution of gas ceases, add slowly and with stirring a cold saturated soln. of $[Co(NH_3)_6]Cl_3$ in excess. After 5 min. filter off the yellow crystalline ppt. after washing 2 or 3 times by decantation with a 0·1% soln. of $[Co(NH_3)_6]Cl_3$. Transfer the ppt. completely to the crucible and wash it with a soln. containing 4 ml of ethanol, 3 ml of water and 1 or 2 drops of the 0·1% soln. of $[Co(NH_3)_6]Cl_3$. Finally wash the ppt. with 1 or 2 ml of abs. ethanol in small portions, and 6 to 8 ml of ether. Dry in a vacuum desiccator and weigh as $[Co(NH_3)_6][Co(NO_2)_6]$. In determining up to 0·0366 g of Co the max. error was 0·08 mg. The method may be used on a micro scale. C. D. Kopkin

941. **Colorimetric and polarographic methods for the determination of cobalt and the indirect determination of potassium.** R. Ralea and F. Modreanu (Inst. of Chem. Research, P. Poni Acad., Iaşi, Romania). *Rev. Chim., Acad. R.P.R.*, 1958, **3** (1), 31–45 (in French).—When $Co^{2+}$ are oxidised with $Na_2O_2$ in the presence of sulphosalicylic acid (**I**) the colour formed is brown, yellow - brown, yellow - green or intense green as the concn. of **I** increases, with little change of extinction; at least 20 ml of 0·1 *M* **I** is necessary to develop the green colour with 0·91 mg of Co, 10 to 20 mg of $Na_2O_2$ being required. The colour is stable only in strongly alkaline soln. To determine Co colorimetrically, to the soln. containing up to 1·6 mg of Co add 5 ml of a saturated Na sulphosalicylate soln., 10 to 20 mg of $Na_2O_2$ and, after no more O is evolved, 0·1 to 0·2 mg of $Na_2SO_3$. Make up to 50 ml and measure the extinction with a blue filter in a 10-mm cell, with water containing 5 ml of the sulphosalicylate soln. in 50 ml as comparison soln. Construct a calibration curve. In determining 0·91 mg of Co, the presence of up to 10 mg of Ni or Zn, or up to 2 mg of Cd, does not cause interference; Fe, Cu, V, Mn and U interfere. To determine Co polarographically, use is made of the reduction of the green compound, or the similar compound obtained with salicylic acid. The basal soln. used are (*i*) saturated Na sulphosalicylate soln. (4 ml), 2 *M* Na acetate (1·5 ml), $Na_2O_2$ (10 to 40 mg), *M* $Na_2SO_3$ (2 ml) and 2% gelatin soln. (0·5 ml) (for a total vol. of 10 ml), or (*ii*) *M* Na salicylate (4 ml), 2 *M* Na acetate (1·5 ml), $Na_2O_2$ (10 to 40 mg), *M* $Na_2SO_3$ (2 ml) and 0·2% gelatin soln. (0·5 ml) (for a total vol. of 10 ml). The Co is reduced at 0 V, polarography being carried out 20 to 30 min. after preparing the soln. With the salicylate reagent, the wave height is 2·5 times that for the sulphosalicylate reagent; the wave height is proportional to concn. of Co. With sulphosalicylate the error in determining 4·55 mg of Co is generally $\pm$2%. No influence on wave height is shown by Ni, Cd and Zn. The presence of Fe or Cu causes a decrease in wave height. Manganese is also reduced at 0 V and so interferes. Potassium may be determined indirectly after pptn. as cobaltinitrite.
C. D. Kopkin

942. **The study of rhodium(III) complexes by paper electrophoresis and ion-exchange chromatography. II.** M. Lederer (Inst. du Radium, Paris, France). *J. Chromatography*, 1958, **1** (3), 279–284.—The action of acids on $Rh(OH)_3$ has been studied. A cation of low mobility, which is unstable and decomposes into several complexes, is formed in HCl, HBr and $HNO_3$ soln., but not in $H_2SO_4$ soln. All of the complexes have low $R_F$ values and may be suitable for the paper-chromatographic separation of $Rh^{III}$ from $Pt^{IV}$ and $Pd^{II}$. A. R. Rogers

943. **Studies on the applications of organic cations as analytical reagents. I. NN-Dimethylpiperidinium iodide as a reagent for palladous ions.** Ch'ing-Hsin Yung and K'uei Wang. *Acta Chim. Sinica*, 1957, **23** (4), 250–254.—NN-Dimethylpiperidinium iodide is recommended as a reagent for $Pd^{2+}$ with which it forms a black or brown ppt. The sensitivity is 0·2 $\mu$g (1 in $2 \times 10^5$) on a spot plate and 0·006 $\mu$g (1 in $2 \times 10^5$) on filter-paper. By the addition of a large amount of phosphate 2 $\mu$g of $Pd^{II}$ can be detected in the presence of 40 $\mu$g of Pt; $Bi^{3+}$ and $Hg^{2+}$ interfere. Sci. Abstr. China

944. **2-Mercaptobenzothiazole as an analytical reagent. Estimation of palladium.** A. K. Majumdar and M. M. Chakrabartty (Jadavpur Univ., Calcutta-32, India). *Z. anal. Chem.*, 1958, **162** (2), 96–100 (in English).—The reagent is selective for the determination and separation of Pd as $Pd(C_7H_4NS_2)_2$ from alkali and alkaline-earth metals, Mg, $Fe^{3+}$, Cr, Th, Zr, Ti, $UO_2^{2+}$, Be, the rare-earth metals, $Ce^{3+}$, $Ce^{4+}$, Zn, Mn, Co, Ni, Pb, Bi, $Sb^{3+}$, $As^{3+}$, $Sn^{4+}$, Tl, $Cu^{2+}$, Cd, $Ir^{4+}$, $Rh^{3+}$, $Ru^{3+}$, $Os^{4+}$, $CrO_4^{2-}$, $MoO_4^{2-}$, $WO_4^{2-}$, $VO_3^-$, $PO_4^{3-}$ and $AsO_4^{3-}$ at pH 6 to 8 in the presence of EDTA and tartaric or citric acid; $Pb^{2+}$, $Ag^+$ and $Hg^{2+}$ are kept in soln. with KI. Separation from $Au^{3+}$ is effected by complexing the Au with thiosulphate. The red $Pd(C_7H_4NS_2)_2$ is diamagnetic and is stable up to 388°. It may be weighed after drying or dissolved in excess of cyanide and back-titrated with $AgNO_3$ soln. The coloration and pptn. limits are 0·04 $\mu$g per ml and 0·2 $\mu$g per ml, respectively. D. F. Phillips

945. **2-Mercaptobenziminazole as an analytical reagent. Estimation of palladium.** A. K. Majumdar and M. M. Chakrabartty (Jadavpur Univ., Calcutta-32, India). *Z. anal. Chem.*, 1958, **162** (2), 101–105 (in English).—Palladium is pptd. quant. by the reagent at pH 5·0 to 10·1 as the red cryst. compound $Pd(C_7H_5N_2S)_2$, which is diamagnetic and is stable up to 447°. In the presence of a large excess of tartrate or citrate and EDTA, and, at a pH between 6 and 8, Pd can be separated from the alkali and alkaline-earth metals, Mg, $Fe^{3+}$, Cr, Th, Zr, Ti, $UO_2^{2+}$, Be, $Ce^{3+}$, $Ce^{4+}$, the rare-earth metals, Zn, Mn, Ni, Co, Pb, Bi, As, $Sb^{3+}$, $Sn^{4+}$, $Tl^+$, $Cu^{2+}$, Cd, $Ir^{4+}$, $Rh^{3+}$, $Ru^{3+}$, $Os^{4+}$, $CrO_4^{2-}$, $MoO_4^{2-}$, $WO_4^{2-}$, $VO_3^-$, $PO_4^{3-}$ and $AsO_4^{3-}$; Pb, Ag and $Hg^{2+}$ are kept in soln. by KI, and a small amount of $Au^{3+}$ by thiosulphate. The complex is either weighed after drying at 110° or dissolved in standard cyanide soln. and back-titrated with $AgNO_3$ soln.
D. F. Phillips

946. **Critical evaluation of colorimetric methods for the determination of platinum metals. II.** F. E. Beamish and W. A. E. McBryde (Univ. of

Toronto, Ontario, Canada). *Anal. Chim. Acta*, 1958, **18** (6), 551–564.—Some general considerations are presented together with a review of colorimetric methods for Pt, Pd, Ir, Rh, Os and Ru published between May 1953 and March 1957.
H. N. S.

947. **X-ray fluorescence analysis of ores and minerals for uranium, thorium, lead, tantalum, hafnium, niobium, zirconium, yttrium and strontium.** K. I. Narbutt and I. D. Bespalova. *Zavod. Lab.*, 1958, **24** (5), 617–619.—A universal focusing X-ray spectrometer (Narbutt, *Izv. Akad. Nauk, SSSR, Ser. Fiz.*, 1953, **17**, 249, and 1956, **20**, 136), a Geiger - Müller counter, and a special method, which is described, of weighing and preparing the sample, are used. G. S. Smith

948. **Activation analysis for heavy elements in stone meteorites.** G. W. Reed, K. Kigoshi and A. Turkevich (Argonne Nat. Lab., Lemont, Ill.). *U.S. Atomic Energy Comm.*, Rep. A/CONF.15/P/953, 1958, 11 pp.—This analytical method has been applied to the determination of U in meteorites. It has been found that four chondritic meteorites contain, within about 10%, the same amount of U ($1{\cdot}1 \times 10^{-8}$ g per g); the amount of U in iron meteorites is at the limit of sensitivity of the method with the neutron fluxes available ($10^{-11}$ to $10^{-12}$ g). The results on both stone and iron meteorites are factors of 10 to 100 lower than most previous determinations. Nucl. Sci. Abstr.

949. **Spectrographic analysis of slags.** B.I.S.R.A. Methods of Analysis Committee. *J. Iron St. Inst.*, 1958, **189** (1), 49–55.—Pelleting, tipping, dry powder and solution techniques were investigated in an attempt to formulate a method that would give the complete analysis with an accuracy of $\pm$ 3% in 30 min. Detailed attention is given to the making of powdered samples, homogeneity of the final melt, effect of the fusion-mixture ratio, and polarity of the sample, and, for solution techniques, the effect of electrode shape and preliminary treatment. A recommended solution technique is given, but no attempt has been made to reduce the operation time of 30 to 45 min. to obtain a triplicate analysis of a slag sample. J. W. O. Pyemont

950. **Correlation of physical constants and chemical structure. Graphical statistical methods for the identification of mineral oils, glass, silicones, fatty oils and catalysts. III. The composition of glasses and silicones.** H. I. Waterman, C. Boelhouwer and J. Cornelissen (Technol. Univ., Delft, The Netherlands). *Anal. Chim. Acta*, 1958, **18** (5), 469–480 (in English).—The specific refraction of sodium glasses and of potassium glasses is in each case in linear relation to the composition (mol. % $SiO_2$) and is independent of the temperature of previous treatment. From graphs of this relation the composition of these glasses can be determined by measurement of the refractive index and density. Methods of measurement of these constants are described. The temperature dependence of the kinematic viscosity of molten glasses and of silicones is discussed. The specific refraction and specific parachor of cyclic silicones are independent of the degree of polymerisation. This suggests that the changes caused in glass by thermal treatment may be connected with polymerisation and depolymerisation. G. Burger

951. **Application of EDTA titrations to the analysis of electroplating baths.** E. Bauman (Zavod za Anorg. Kem. Tehnol., Tehnol. Fak., Zagreb, Yugoslavia). *Kem. u Ind., Zagreb*, 1958, **7** (6), 141–146.—The use of EDTA for determinations of Zn, Cu, Cd, Ni, Sn, Pb and Mg was studied, and techniques for the estimation of each of these metals in the main types of electroplating baths are described. Results are tabulated and indicate a remarkably high degree of accuracy.
A. Grochowski

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*See also Abstracts*—**1008**, Determination of phosphates in biological materials. **1092**, Determination of Bi. **1127**, Determination of U in water. **1128**, **1129**, Determination of radionuclides in water. **1133**, Determination of $^{90}Sr$ in soil. **1145**, Control of $CO_2$ in gases.

## 3.—ORGANIC ANALYSIS

*Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather, explosives.*

952. **Use of polarography in organic analysis.** G. E. Penketh (Phys. Chem. Div., Res. Dept., I.C.I., Billingham). *J. Polarograph. Soc.*, 1958, (3), 42–46.—A review of the applications of polarography in organic analysis with a short description of the methods used. N. E.

953. **Application of polarography in organic chemistry.** I. A. Korshunov. *Zavod. Lab.*, 1958, **24** (5), 543–548.—A review of papers published in the years 1954 to 1956. (101 references.)
G. S. Smith

954. **Method for the determination of phosphorus in organic compounds.** R. A. Chalmers and D. A. Thomson (Chem. Dept., The Univ., Old Aberdeen, Gt. Britain). *Anal. Chim. Acta*, 1958, **18** (6), 575–577.—A method for the determination of up to 1600 $\mu$g of P is described. Decompose the sample (3 to 10 mg) with 1 ml of conc. $H_2SO_4$ and a few drops of conc. $HNO_3$. After evaporation to fumes and dilution, transfer the soln. to a calibrated flask, and add a dil. $H_2SO_4$ soln. of ammonium molybdate and $(NH_4)_2SO_4.FeSO_4$ soln. After dilution to 100 ml, set the soln. aside for 15 min. and measure the optical density at 750 m$\mu$ against a blank containing the reagents. Variables affecting colour development are discussed. The method is rapid and sufficiently accurate for normal purposes.
F. L. Selfe

955. **Sub-micro methods for the analysis of organic compounds. III. Determination of N-methyl groups and the simultaneous determination of alkoxyl and N-methyl groups.** R. Belcher, M. K. Bhatty and T. S. West (Chem. Dept., The Univ., Birmingham, England). *J. Chem. Soc.*, 1958, 2393–2396.—A modification of Herzig and Meyer's method for the N-methyl group (*Monatsh.*, 1894, **15**, 613; 1895, **16**, 599; 1897, **18**, 379) is described. A decomposition flask containing glass beads to increase its internal surface is connected to a flask, then through a reflux condenser to a scrubber and

an absorption flask. *Procedure*—About 50 μg of weighed sample is introduced into the decomposition flask with 20 mg of $NH_4I$, 0·05 g of phenol, 2 drops of 2% gold chloride soln., and 0·5 ml of iodine-free HI, added in that order. The scrubber is charged with 8 ml of sodium antimonyl tartrate soln., and a silica-wool plug soaked with the same soln. is put in the neck of the condenser. The absorber is charged with 1 ml of Na acetate soln. in glacial acetic acid and ≃0·05 ml of Br. A regulated stream of N is passed (1 bubble per sec. in the scrubber) and the flasks are heated for 3 hr. so that the decomposition flask reaches 100° in 0·25 hr. and 360° in 2 hr., and the reflux flask reaches 50° in 0·25 hr. and 150° in 1·5 hr. At the end of 3 hr., the flasks are cooled to room temp., the absorber and scrubber are disconnected and the HI in the reflux flask is returned to the decomposition flask. The procedure is repeated twice (only once more for a blank) with a fresh absorber. The contents of the absorber are titrated with 0·01 *N* $Na_2S_2O_3$. If methoxyl groups are present they are absorbed as methyl iodide and determined first by heating the flasks to 150° and 50°, respectively, for 1 hr.

E. J. H. Birch

956. **Separation and analysis of mixtures of chloro derivatives of methane by a chromatographic method.** D. A. Vyakhirev and L. E. Reshetnikova. *Zhur. Prikl. Khim.*, 1958, **31** (5), 802–805.—Elution with N is carried out on a liquid paraffin - kieselguhr column heated by a movable furnace, and the chromatograms, obtained by a thermal-conductivity method, are recorded automatically. Analysis of a mixture of $CH_3Cl$, $CH_2Cl_2$, $CHCl_3$ and $CCl_4$ is carried out in 20 to 40 min. G. S. Smith

957. **Determination of peroxides in ether distillate and ethereal uranyl nitrate solutions.** Ministry of Supply, Chemical Inspectorate (Springfields Works, nr. Preston, Lancs.). U.K.A.E.A. Report SCS-M-119, 1958, 5 pp.—Uranyl nitrate soln. are first washed with water. The sample is shaken with $NH_4SCN$ and $(NH_4)_2SO_4.FeSO_4$ soln. The concn. of the $Fe^{III}$ complex produced is determined by visual comparison with standard soln. containing $KMnO_4$ and $K_2Cr_2O_7$. The method is suitable for samples with contents of peroxides from 1 to 10 p.p.m. G. J. Hunter

958. **Quantitative determination of organic per-compounds in mixtures.** L. Horner and E. Jürgens (Organ.-chem. Inst., Univ., Mainz, Germany). *Angew. Chem.*, 1958, **70** (9), 266–268.—After a brief review of methods previously developed for the identification and determination of organic per-compounds, new procedures are described for the quant. analysis of these compounds in mixtures. Both diphenyl sulphide and triethyl- (or triphenyl-) arsine react specifically with certain groups of per-compound and by combining these reactions with standard iodimetric and acidimetric procedures it is possible to determine the composition of mixtures of four groups of compound, *viz*, dialkyl peroxides (**I**), alkyl hydroperoxides (**II**), per-acids (**III**) and diacyl peroxides (**IV**). Experimental details are given and the methods of calculation outlined for the analysis of all binary, tertiary and quaternary mixtures of **I**, **II**, **III** and **IV**. The various procedures which can be applied to each type of per-compound are tabulated. S. M. Marsh

959. **Analysis of polyoxypropylene glycols.** E. H. Vogelenzang and D. J. Stöver (Farm. Lab., Rijksuniv., Leiden, Holland). *Pharm. Weekbl.*, 1958, **93** (11), 550–555.—Acetic anhydride in pyridine causes hydrolysis of ether bonds influenced by methyl groups, so that results for hydroxyl-group determination are high. Pesez's propionic anhydride method (*cf. Anal. Abstr.*, 1955, **2**, 1237) gives satisfactory results after correction for the water content (Karl Fischer). P. Rentenaar

960. **Colorimetric determination of carbohydrates with dinitrosalicylic acid.** V. V. Kozlov and V. N. Khrustaleva. *Sb. Nauch. Rabot Mosk. Inst. Nar. Khoz.*, 1957, (10), 353–372; *Ref. Zhur., Khim.*, 1958, Abstr. No. 50,066.—A method for the determination of sugars by the use of 3:5-dinitrosalicylic acid is described. W. Roubo

961. **Estimation of formates by oxidation with mercuric chloride.** Sameer Bose (Chem. Dept., Mahakoshal Mahabidyalaya, Jabalpur). *J. Indian Chem. Soc.*, 1958, **35** (5), 320–322.—A method, applicable in the presence of salts of other organic acids, is described for the volumetric and gravimetric determination of formate. The precision of the volumetric method is 0·5% or better. An iodimetric method, suitable for the micro-determination of amounts as small as 1 mg of formic acid, is also described. I. Jones

962. **Improved micro-diffusion procedure for the determination of lactic acid.** H. Ryan (Dept. of Biochem. and Pharmacol., Univ. Coll., Dublin). *Analyst*, 1958, **83**, 528–531.—The micro-diffusion is effected in Conway No. 1 units. To prepare the calibration graph, 2 ml of semicarbazide reagent (0·0067 *M* in a phosphate buffer soln. of pH 7) is placed in the centre chamber and 0·4 ml of satd. $Ce(SO_4)_2$ soln. (in 6 *N* $H_2SO_4$) in the outer chamber and then 2 ml of standard lactic acid soln. in the concn. range 10 to 70 μg per ml. The units are sealed with paraffin wax - petroleum jelly fixative and, after mixing the contents of the outer chamber, are incubated at 37° for 2 hr. From the centre chamber of each 1 ml is removed, diluted to 10 ml and the extinction is measured at 224 mμ against a blank soln., with water in place of the lactic acid soln. The graph is linear for the stated range. To determine lactic acid in blood, a 2-ml portion of blood is centrifuged with 2 ml of 15% $HClO_4$ and 7 ml of water. From a measured aliquot the excess of $HClO_4$ is removed by pptn. with 40% KOH soln., and 2 ml of the supernatant liquid is treated as described for the calibration, the blank now being 0·4 ml of 6 *N* $H_2SO_4$ in place of the $Ce(SO_4)_2$ soln. Application of the method to determination of lactic acid in rat-leg muscle is described.

A. O. Jones

963. **Spectroscopic determination of lactic acid.** G. di Modica and E. Angeletti (Turin Univ., Italy). *Ric. Sci.*, 1958, **28** (7), 1485–1488.—The lactic acid (0·14 to 1·3 mg) is oxidised with I - KI soln. in alkaline medium to $CHI_3$, which is extracted with $CHCl_3$ and measured by u.v. spectroscopy. The max. at 347 mμ is used and correction is made for the slow photochemical decomposition of the soln.

L. A. O'Neill

964. **Oxidation of some α-hydroxy acids and of mannitol with lead(IV) acetate and potassium periodate.** A. Berka and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague). *Chem. Listy*, 1958, **52** (5), 930–935.—The velocity of the oxidation of tartaric, gluconic and mandelic acids in aq. soln. of pH 4·8 by means of $Pb^{IV}$ acetate (**I**) and $KIO_4$ (**II**) has been studied by direct potentiometric titration, or

by indirect titration when the excess of the reagent can be determined with quinol (for **I**) or with $FeSO_4$ ($KIO_4 \rightarrow KIO_3$), the excess of which can be titrated visually with $KIO_4$, with diphenylamine as indicator. The direct titration of the acids with **I** is possible, but some difficulties occur as a result of the further oxidation of the aldehyde or formic acid produced. If an excess of the reagent is used, $CO_2$ and $H_2O$ are the principal products of the oxidation. For the quant. determination of mannitol, or tartaric or gluconic acid the indirect method with $KIO_4$ was found to be the most suitable. *Procedure*—Dissolve the sample (about 2 mg) in $H_2O$ (5 ml), add acetate buffer soln. (pH 4·8) (glacial acetic acid half neutralised with 30% NaOH soln.) (2 ml) and 10 ml of 0·01 *M* $KIO_4$. Set aside for 4 hr., add conc. $H_2SO_4$ (1·5 ml) dropwise, cool, add 10 ml of 0·02 *N* $FeSO_4$ and titrate with 0·01 *M* $KIO_4$. Diphenylamine as indicator must be added at the beginning of the titration and another portion near the end-point. A blank determination is carried out. J. Zýka

965. **Separation of aliphatic amines by paper chromatography and paper electrophoresis.** E. J. Herbst, D. L. Keister and R. H. Weaver (Maryland Univ., Baltimore, U.S.A.). *Arch. Biochem. Biophys.*, 1958, **75** (1), 178–185.—Paper chromatography is carried out on Whatman No. 1 paper by the descending technique for 16 hr. at room temp. with 2-(2-ethoxyethoxy)ethanol (**I**) - propionic acid - water (14:3:3) saturated with NaCl or with a similar mixture containing 2-ethoxyethanol in place of **I**. For certain separations better results are obtained by electrophoresis on Whatman No. 1 paper treated with 0·05 *M* sulphosalicyclic acid at pH 4·0 or with 0·03 *M* citric acid at pH 6·5. Migration distances are given for 16 mono-, di- and poly-amines of biological interest. The compounds are detected with ninhydrin or by a bio-autographic technique based on the growth response of *Haemophilus parainfluenzae* to certain amines.

W. H. C. Shaw

966. **Coulometric titrations with uni- and bivalent mercury. Determination of secondary amines and mercaptans.** E. P. Przybylowicz and L. B. Rogers (Res. Lab., Eastman Kodak Co., Rochester 4, N.Y., U.S.A.). *Anal. Chim. Acta*, 1958, **18** (6), 596–606.—The titration of aliphatic amines and mercaptans with electrolytically generated $Hg^{2+}$ reagent is reported. Secondary amines are titrated after conversion into the corresponding dithiocarbamic acids by reaction with $CS_2$. Primary amines interfere and are removed by reaction with salicylaldehyde. Tertiary amines do not interfere. A $3 \times 10^{-4}$ *F* soln. of a secondary amine has been titrated with an error $< 1\%$ and a $3 \times 10^{-5}$ *F* soln. with an error $< 2\%$. The titration of cysteine has been studied briefly. Amounts of cysteine of $\not< 1{\cdot}29$ mg in 100 ml of soln. are determined with an error $< 1\%$ and smaller amounts, down to 0·26 mg in 100 ml, with an error of $\simeq 3\%$.

F. L. Selfe

967. **Studies on acyl *iso*thiocyanates. IV. *p*-Bromobenzoyl *iso*thiocyanate as reagent for identification of amines.** Yao-Tso Chen and Yin-Haw Nee. *Acta Chim. Sinica*, 1957, **23** (4), 280–286.—The *p*-bromobenzoyl *iso*thiocyanates of amines are cryst. deriv. that have higher m.p. and are more easily purified than the benzoyl *iso*thiocyanates or the *m*-nitrobenzoyl *iso*thiocyanates. Twenty-four deriv. of various amines have been examined.

Sci. Abstr. China

968. **Determination of biphenyl [diphenyl] in the presence of polyphenyls. Water solubility method.** L. Silverman and M. E. Shideler (Atomics International, Canoga Park, Calif., U.S.A.). *Anal. Chim. Acta*, 1958, **18** (6), 540–546.—Diphenyl is determined in a mixture of polyphenyls by extracting the mixture with water at 55° and determining the extinction at 247 to 250 mμ of the aq. extract. The solubility of the other polyphenyls is less than that of diphenyl ($\simeq$ 9 mg per litre) to such an extent that 0·1% of diphenyl can be detected in *p*-terphenyl. H. N. S.

969. **Detection of aromatic aldehydes.** E. Sawicki, T. Stanley and T. Hauser (Taft San. Engng Center, U.S. Dept. of Health, Education and Welfare, Cincinnati, Ohio, U.S.A.). *Chemist Analyst*, 1958, **47** (2), 31–32.—The method is based on the reaction of an aldehyde with trifluoroacetic acid to form an arylidene di(trifluoroacetate), which is condensed with a hydrocarbon (fluoranthene) to form a diarylmethane-type dye. As described, the method is suitable for the detection of polynuclear aromatic aldehydes, and could probably be extended to include other types of compound by replacement of the fluoranthene or by using an aldehyde as reagent for the detection of aromatic hydrocarbons, ethers, sulphides, etc. *Procedure*—Mix fluoranthene (5% soln. in $CH_3Cl$) (1 ml) with an equal vol. of a $CH_3Cl$ soln. thought to contain the aromatic aldehyde. Add trifluoroacetic acid (0·8 ml) and mix thoroughly. Allow to stand for 5 min. and dilute to 10 ml with trifluoroacetic acid. A positive reaction is usually indicated by the development of a blue or green colour. G. S. Roberts

970. **Preliminary classification of some naturally occurring hydroxycinnamic acids through their ultra-violet spectra.** G. K. Sutherland (Hawaii Univ., Honolulu). *Arch. Biochem. Biophys.*, 1958, **75** (2), 412–417.—The technique proposed is based on the u.v. spectra given by the different classes of hydroxycinnamic acids, as free acids or as esters, in acid, alkaline and ethanol solution at concn. of about $5 \times 10^{-5}$ *M*. The compounds studied include caffeic, ferulic, *p*-coumaric and chlorogenic acids and 7-hydroxycoumarin.

W. H. C. Shaw

971. **Vanillin as a reagent in the determination of primary arylamines.** E. N. Deeb (Veterans Admin. Hosp., Pittsburgh, Pa.). *Drug Standards*, 1958, **26** (5), 175–177.—Vanillin reacts with primary aromatic amines at pH 0·5 to give a yellow colour, with max. absorption at 400 mμ, which is stable for 45 min. (*cf. Anal. Abstr.*, 1955, **2**, 2508). Aliphatic and secondary aromatic amines do not interfere. A. R. Rogers

972. **Determination of small amounts of aromatic amines in the presence of *iso*cyanates formed therefrom in air and dilute solutions.** W. Pilz (Farbenfabrik. Bayer A.-G., Leverkusen, Germany). *Mikrochim. Acta*, 1958, (4), 545–552 (in German).—Aromatic *iso*cyanates react with NN′-dimethylethylenediamine (**I**) to form ureas which are not further decomposed to amines with *N* HCl. Aromatic amines present in the *iso*cyanates do not take part in the reaction and can be determined by diazotising and coupling with N-1-naphthylethylenediamine; the extinction is measured with suitable filters. The sample of air is passed through 10 ml of a 0·5% soln. of **I** in dimethylformamide. The resulting soln. is diluted with 10 ml of *N* HCl and

diazotised. For samples in soln., 5 ml is added to the soln. of **I** and shaken for 10 min. Aliphatic amines do not interfere. D. F. PHILLIPS

973. **Quantitative determination of *o*- and *p*-ethylnitrobenzene.** J. Neumann, V. Štěpánková and Z. Aunický (Inst. Inorg. Technol., High School of Chem. Technol., Prague). *Chem. Průmysl*, 1958, **8** (5), 244–246.—The method is based on the quant. oxidation of *p*-ethylnitrobenzene (**I**) to *p*-nitrobenzoic acid (**II**) with $CrO_3$ in boiling glacial acetic acid and gravimetric determination of **II**, which is sparingly sol. in water. Only minute amounts of *o*-ethylnitrobenzene (**III**) react under these conditions and the *o*-nitrobenzoic acid formed is sol. in water and does not therefore interfere. *Procedure*—Dissolve the mixture of **I** and **III** (2 g) in glacial acetic acid (100 ml), boil under reflux and add gradually a satd. aq. soln. of $CrO_3$ (10 g of $CrO_3$ in 6 ml of $H_2O$) (10 ml). After 1 hr., cool the product and dilute it with $H_2O$ at 15° to 450 ml. Separate the **II** on a glass filter (S3), wash it with $H_2O$ (50 ml), dry at 100° and weigh. Results are accurate to within ± 1%. A mixture of *p*-nitroacetophenone with its isomers can be determined in the same way. J. ZÝKA

974. **Volumetric and polarographic determination of N-methyl-N-nitrosourea and investigation of its stability.** F. Jančík, B. Kakáč, V. Vaníček and M. Vrublovská (Res. Inst. Pharm. and Biochem., Prague). *Chem. Listy*, 1958, **52** (5), 909–914.—The volumetric determination of N-methyl-N-nitrosourea (**I**) is based on the titration of the excess of NaOH after hydrolysis of **I**. For the polarographic determination, the selective reduction of the nitroso group of **I** has been used; **I** in pH 2 to 8 yields a well-developed polarographic wave, the $E_{\frac{1}{2}}$ of which depends on pH; a small catalytic wave which appears in acid soln. can be suppressed by gelatin. *Volumetric procedure*—To the sample (150 to 200 mg) in a 150-ml flask add, with stirring, 25 ml of 0·1 *N* NaOH and mix till no more diazomethane is liberated. Add phenolphthalein as indicator and titrate with 0·1 *N* $H_2SO_4$. *Polarographic procedure*—Dissolve the sample (40 mg) in $H_2O$ (100 ml) and to 0·2 to 1 ml in a 10-ml flask add 5 ml of buffer soln. (pH 5) and 0·2% gelatin soln. (0·5 ml) and make up to vol. with $H_2O$. Remove O with a stream of N and register the wave from − 0·4 V. The soln. of **I** are unstable and decompose after 30 min. The method described has been used for the investigation of the stability of **I** in the solid state and in soln. at different temp. and pH. Existing data on the stability of **I** are criticised. J. ZÝKA

975. **Analysis of triphenyltin compounds.** R. Bock, S. Gorbach and H. Oeser (Farbwerke Hoechst A.-G., Frankfurt/M.-Höchst, Germany). *Angew. Chem.*, 1958, **70** (9), 272.—The use of triphenyltin compounds in plant protection makes it necessary to develop methods for the determination of these compounds and their decomposition products. Mixtures of triphenyltin, diphenyltin and inorganic tin compounds (except complexes with organic acids) may be analysed as follows. A weakly acidic (0·2 to 0·3 *N* in $H_2SO_4$) aq. soln. of the mixture is treated with a mixture of disodium tartrate and NaOH so that it becomes about 0·05 *N* in NaOH and 0·1 *M* in disodium tartrate. Triphenyltin hydroxide then separates and is extracted with $CHCl_3$ (2 × 10 ml). The extract is then shaken again with the tartrate - NaOH mixture and the aq. phase is returned to the bulk of the aq. layer which contains the tartrate complexes of diphenyltin and inorganic tin compounds. This aq. layer is neutralised with dil. $H_2SO_4$, and 7 ml of 0·5% tetramethylenedithiocarbamate soln. and 10 ml of 0·5% EDTA (disodium salt) soln. are added. The pH of the resulting soln. is adjusted to 8·0 (glass electrode) with a borate buffer. Diphenyltin complexes separate and are removed by extraction with $CHCl_3$ in a manner similar to that used for the removal of the triphenyltin complexes. The $CHCl_3$ extracts obtained are then evaporated and treated with a $H_2SO_4$ - $HNO_3$ mixture. The amount of tin in the residues is determined by conventional methods. Results obtained on synthetic mixtures are tabulated to indicate the accuracy of the method. S. M. MARSH

976. **Technological problems of continuous gas chromatography (hypersorption).** P. Benedek, L. Szepesy and Z. Nagy (Hungarian Inst. for Oil and Natural Gas). *Magyar Kém. Lapja*, 1958, **13** (4), 117–123.—Pilot-plant-scale experiments and measurements are described for the separation of natural-gas products by a continuous counter-current adsorption - desorption column of activated carbon. The separator unit (30 metres high) consists of a heated desorber (lower part), rectifying adsorber (diam. 20 cm) and cooling section (top), and is equipped with injection and reflux apparatus and instruments. Activated carbon (200 to 250 kg per hr.) is circulated by pneumatic methods. The apparatus has been tested with a mixture of 87·7% of N, 5·4% of $CO_2$ and 6·9% of acetylene. If rectified side-product removing is employed, 100% pure N is obtained as top product, 99·2% pure $CO_2$ as side-product and 98·0% pure acetylene as bottom product. Nitrogen of 99% purity can be separated from a mixture of 87% of N, 2·8% of ethane, 7·4% of propane and 2·8% of butane. The problems of construction, quality of activated carbon, heating, hydraulics and instrumentation are discussed. G. SZABO

977. **Analysis of hydrocarbons. V. Analysis and evaluation of crude oils. 1. Groundwork, apparatus, test methods.** H. Grosse-Oetringhaus (Heimfelder Str., 69, Hamburg-Harburg, Germany). *Erdöl u. Kohle*, 1958, **11** (5), 300–304.—Details of design of a laboratory apparatus, now standardised in Germany (DIN 51567, 1957), for fractionating petroleum, at 0·01 mm of Hg, into any desired cuts down to a residue softening at ≮40° (ring and ball), are described. A. R. PEARSON

978. **Determination of phosphorus in gasoline.** E. R. Fett and G. Matsuyama (Union Oil Co. of Calif., Brea, U.S.A.). *Chemist Analyst*, 1958, **47** (2), 32–34.—The modified molybdenum blue method of Berenblum and Chain (*Biochem. J.*, 1938, **32**, 286, 295) has been adapted to the determination of organophosphorus additives in petrol. G. S. ROBERTS

979. **Semi-automatic apparatus for the determination of tetraethyl-lead in fuels.** G. Zuliani and L. Orlandi (Ist. Chim. Fis., Padua, Italy). *Ric. Sci.*, 1958, **28** (7), 1480–1484.—Apparatus and procedure are described. The tetraethyl-lead is decomposed with conc. HCl, and the Pb, in HCl soln., is determined polarographically in the presence of Cd as pilot ion. L. A. O'NEILL

980. **Correlation of physical constants and chemical structure. Graphical statistical methods for the identification of mineral oils, glass, silicones, fatty oils and catalysts. I. Introduction. II. Mineral oils and related hydrocarbon mixtures.** H. I. Waterman, C. Boelhouwer and J. Cornelissen (Technol. Univ., Delft, The Netherlands). *Anal. Chim. Acta*, 1958, **18** (5), 395–468 (in English).—By means of graphical statistical methods, mixtures too complicated for their components to be separated can be characterised by the correlation existing between physical constants and the structure of a hypothetical molecule representing the proportions of the various structural groups present in the mixture. Known methods of characterising mineral oils by carbon type and ring analysis and the selective hydrogenation of aromatic components are treated in detail. Graphs and nomograms are presented which represent the correlation between physical constants and between these and structural composition. The construction and use of these diagrams are explained. From them the average number of each ring type, the proportion of the total C which is combined in each ring type, the mean mol. wt. and some constants can be estimated. Among the methods treated are the *n–d–M* method and the *v–n–d* method. Among the constants are the temp. dependence of viscosity, ultrasonic sound velocity and the surface tension. The degree of branching cannot be quantitatively determined, but some comparative estimates may be made with the aid of the parachor and the magneto-optical rotation. Aromatic components, a high concn. of which invalidates these relationships, can be sharply separated from naphthenes and paraffins and roughly separated into mono-, di- and tri-aromatics by percolation through silica gel. Sulphur compounds can be characterised if they can be separated from the hydrocarbons. G. Burger

981. **Correlation of physical constants and chemical structure. Graphical statistical methods for the identification of mineral oils, glass, silicones, fatty oils and catalysts. V. Composition of coal.** H. I. Waterman, C. Boelhouwer and J. Cornelissen (Technol. Univ., Delft, The Netherlands). *Anal. Chim. Acta*, 1958, **18** (5), 493–496 (in English).—The formation of coal from plant material is represented on a diagram of which the axes are H/C and O/C. Any compound composed of C, H and O is represented on this diagram as a point. The products obtained by removal of $CH_4$, $CO_2$ or $H_2O$ from any starting material, *e.g.*, cellulose, are represented by straight lines. The construction of this diagram is explained. Diagrams representing the average mol. structure present at different stages during the conversion of wood into graphite via coal are presented. G. Burger

982. **Determination of cyanogen in coke-oven gases.** S. M. Golyand and N. V. Adon'eva (Sci. Res. Inst. for Industrial and Sanitary Purification of Gases). *Zavod. Lab.*, 1958, **24** (5), 541–542.—The content of $CN^-$, including that of HCN and $(CN)_2$, is determined by absorption in sodium polysulphide soln. and titration of the $SCN^-$ that are formed by Schulek's CNBr method. No interference is caused by the presence of large amounts of $H_2S$ and $CO_2$. The results are calculated as HCN (%). The gas (10 to 12 litres measured over salt soln.) is passed at the rate of 6 to 10 litres per hr. through two absorption tubes each containing 2·5 ml of *M* $Na_2S_3$ and 2·5 ml of water. The combined soln. are treated with 2 *N* $H_2SO_4$ and then 10% aq. $NH_3$ so that the reaction is just alkaline to methyl orange indicator, and the diluted soln. (50 ml) is boiled, then cooled, mixed first with 10 ml of 2 *N* $H_2SO_4$ and then with bromine water until the yellow colour persists, and set aside in a stoppered flask for 2 to 3 min. Phenol or formic acid is added to remove Br, 1 g of KI is added after 15 min., and the titration with 0·1 or 0·05 *N* $Na_2S_2O_3$ is carried out after a further 15 min. in the dark.

G. S. Smith

983. **Determination of reducing sugars in wood hydrolysates and sulphite liquor by paper chromatography.** I. Z. Emel'yanova and T. A. Batrakova (All-Union Sci. Res. Inst.). *Zhur. Anal. Khim.*, 1958, **13** (1), 142–147.—Separation of the sugars is effected by descending paper chromatography with the solvent ethyl acetate - pyridine - water (5:1:5) at 18° to 22°. The dried chromatograms are sprayed with a soln. of 1·66 g of phthalic acid and 0·92 g of freshly distilled aniline in 100 ml of ethanol. Reducing sugars are determined by a potentiometric titration at the boiling-point of an alkaline cupric tartrate soln. containing potassium ferricyanide with the use of a platinum electrode.

W. Roubo

984. **An absolute method for the determination of vegetable matter in scoured wool.** R. P. Harker (Wool Industries Res. Ass., "Torridon," Headingley, Leeds). *J. Text. Inst., Trans.*, 1958, **49** (5), T227–T231.—Methods for the determination of the vegetable content of scoured wool are discussed, and an absolute method is described in which flotation and density gradient techniques are used. This is applicable only to material mechanically reduced to a fine state, *e.g.*, by a rotary cutter. The complete sample is recovered after separation and without degradation. Results show that duplicates appear to lie within the limits ± 0·5%.

O. M. Whitton

985. **Paper-chromatographic identification of sorbitan fatty acid esters, polyethylene glycol fatty acid esters, and polyethylene glycol sorbitan mono fatty acid esters.** R. Schrepfer and H. Egle (Forschungslab. Nährmittelwerk W. Eiselen, Ulm, Germany). *Z. Lebensmitt Untersuch.*, 1958, **107** (6), 510–512.—The esters, separated from bakery additives by extraction with $CHCl_3$, are saponified by ethanolic KOH. In preparation for chromatographic analysis, unsaponifiable matter and fatty acids are removed from the acidified soln. by extraction with benzene, and inorganic ions are removed from the neutralised soln. by passage over strong cation- and anion-exchange resins. Development is effected in *n*-butanol saturated with water. The dried chromatogram is sprayed with a Pb tetra-acetate reagent. The $R_F$ values of the alcoholic constituents of the esters are tabulated. A 50% ethanolic soln. of glycerol and sorbitol is used as a standard.

P. S. Arup

986. **Error in the sampling of soap and detergent bars for moisture determination.** L. Gildenberg and E. W. Blank (Colgate-Palmolive Co., Jersey City, N.J., U.S.A.). *J. Amer. Oil Chem. Soc.*, 1958, **35** (2), 102–103.—The A.S.T.M. method of sampling soap and combination bars, which permits the bar to be quartered and shavings taken from the freshly cut surfaces, can give results that are not representative of the mean moisture content. Samples taken from 24 points in one bar of soap had moisture

contents varying from 9·10% to 10·97%. Either the whole bar or a symmetrical portion should be taken for analysis. G. Burger

987. **Fluorimetric determination of 7-geranyloxy-coumarin in lemon oil. Analysis of mixtures of grapefruit oil in lemon oil.** S. H. Vannier and W. L. Stanley (Fruit and Veg. Chem. Lab., Agric. Res. Service, U.S. Dept. Agric., Pasadena, Calif., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 432–435.—The method, procedure for which is described, consists in chromatographing mixtures of grapefruit oil and lemon oil on chromatostrips coated with silicic acid, recovering the 7-geranyloxy-coumarin and 5-geranyloxy-7-methoxycoumarin, present respectively, cleaving the geranyloxy groups with acid, extracting the coumarin phenols so produced, and measuring the fluorescence in u.v. light (366 mμ) in alkaline ethanol - water soln. in a photofluorimeter. Results obtained with authentic mixtures were read from standard curves. The values obtained suggest that grapefruit oil contains another fluorescing substance having the same migration rate on the chromatostrips as 7-geranyloxycoumarin. A. A. Eldridge

988. **Analysis of lipsticks by partition chromatography.** M. E. Pietrak and S. H. Newburger (Div. Cosmetics, Food and Drug Admin., Dept. of Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 435–440.—The method described is applicable to both "castor oil" and "oleyl alcohol" types of lipstick. Treatment with trichloroethylene and acetone separates the lakes and fillers; the filtrate is evaporated, the residue is dissolved in heptane, mixed with silane-treated Celite, packed in a tube, and eluted with 50% ethanol, which removes fluorescein dyes and polyethylene glycols. Acetic acid (95%) then elutes castor oil or oleyl alcohol and oil-soluble dyes. Hydrocarbons and waxes, which remain on the column, may be eluted with $CHCl_3$. The procedure for purifying and weighing the fractions is described. The constituents of the fractions are identified by infra-red spectrometry. A. A. Eldridge

989. **Analysis of fat-soluble synthetic colours by reversed-phase paper chromatography.** B. R. Roy and A. R. Sundararajan (Central Food Laboratory, Calcutta). *J. Instn Chem., India*, 1958, **30** (3), 157–160.—Filter-paper discs impregnated with different proportions of medicinal liquid paraffin were used for the circular chromatography of synthetic fat-soluble dyes, with aq. ethanol of various concn. as developer. $R_F$ values were obtained for Sudan 1, Ceres yellow GRN, Ceres yellow 3G, Ceres red, Oil scarlet and nigrosine; these values in general increase with increasing proportion of ethanol in the solvent, and decrease with increasing proportion of liquid paraffin used for impregnating the discs. The constituent colours of 4 mixtures were successfully separated. The method appears promising for identifying and estimating fat-soluble colours in foodstuffs. M. D. Anderson

990. **Detection of reduction products of basic and acidic azo dyes by paper chromatography.** Minya Kitahara, Shoji Miyazaki and Hachiro Iiyama (Osaka Municipal Ind. Res. Inst., Kitaogimachi, Kita-ku). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (2), 189–193.—The dye (0·1 g) is reduced with $SnCl_2$ in HCl and submitted to paper chromatography with 2% HCl and a mixture of butanol and conc. HCl (4 + 1) (*Anal. Abstr.*, 1957, **4**, 2273). Chromium complexes of azo dyes are decomposed with Zn and HCl. The $R_F$ values and the colour reactions of the spots with $NH_3$, $FeCl_3$ or 1-naphthol-4-sulphonic acid are summarised. Silk (0·5 g) dyed with these dyes can be similarly treated to produce aq. soln. of the components, which are submitted to paper chromatography. K. Saito

991. **Analysis of polymers by vapour-phase chromatography.** G. de Angelis, P. Ippoliti and N. Spina (Centro Studio Chimica Generale, Rome). *Ric. Sci.*, 1958, **28** (7), 1444–1450.—The sample is pyrolysed at > 550° in the absence of O and the gases produced are analysed by gas chromatography. A column of silica gel is used for the examination of CO - $CH_4$ - $CO_2$ and columns of Celite - tri*iso*butylene and Celite - Vaseline for $CO_2$ and other hydrocarbons. Polymers give a characteristic pattern of pyrolysis gases, and the behaviour of poly(vinyl acetate), a phenolic and a urea resin, poly(methyl methacrylate), a polyester and various types of polyethylene has been examined. L. A. O'Neill

992. **Rapid determination of polyacrylonitrile [poly(vinyl cyanide)] in coatings by infra-red absorption spectrometry.** M. L. Adams and M. H. Swann (U.S. Army Ordnance, Aberdeen Proving Ground, Md.). *Off. Dig. Fed. Paint, Varn. Prod. Cl.*, 1958, **30**, 646–651.—The content of vinyl cyanide as polymer or copolymer in surface coatings may be determined from the i.r. absorption at 4·48 μ, which is characteristic of the cyanide group. By the base-line technique employed, interference from solvents or other resins is negligible. L. A. O'Neill

993. **The determination of the unsaponifiable matter of tall-oil distillates.** S. T. Bauer, R. E. Price and P. R. Gill (Crosby Chemicals Inc., De Ridder, La., U.S.A.). *J. Amer. Oil Chem. Soc.*, 1958, **35** (3), 120–121.—Diethyl ether and light petroleum are equally satisfactory as extraction solvents in the determination of unsaponifiable matter by either the A.S.T.M. or the A.O.C.S. method. The unsaponifiable matter in the fatty-acid fractions of tall-oil distillates comprises mainly decarboxylated thermal degradation products of rosin and fatty acids. G. Burger

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*See also Abstracts*—**851**, Determination of AgI in photographic films. **950**, Properties of silicones. **1111**, Determination of *iso*propyl alcohol and *sec.*-butyl alcohol in amyl alcohol. **1115**, Polymerisation of drying oils.

## 4.—BIOCHEMISTRY
### INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

#### Biological fluids, animal and vegetable tissues

994. **Application of the nuclear reactor in biochemical analysis. Neutron activation chromatography.** A. A. Benson (Pennsylvania State Univ.). *U.S. Atomic Energy Comm.*, Rep. A/CONF.-15/P/858, 1958, 13 pp.—Activation of paper

chromatograms by slow neutron capture in the nuclear reactor has proved useful in biochemical research. The biological trace elements, Co and V, were separated from ashed tissues and determined by measurement of radioactivity induced on the paper chromatograms. Since all chemical separations are performed before activation, the metastable $^{60m}$Co (11 min.) and $^{52}$V (3·7 min.) were used for the analysis. The $^{31}$P in the intermediates of carbohydrate and lipid metabolism is readily activated on paper chromatograms. The method has been used to obtain accurate and unequivocal analyses of the phosphatides of large animals. The phosphatidyl glycerols have been found in bovine spermatozoa. Neutron activation of sulphur compounds of biochemical interest is indicated.

NUCL. SCI. ABSTR.

995. **An automatic method for colorimetric analysis [of blood].** L. T. Skeggs (Dept. Path., Western Reserve Univ. School of Medicine, Cleveland, Ohio). *Amer. J. Clin. Path.*, 1958, **28** (3), 311–322.—In the method described, samples of whole blood or serum are successively pumped into a flowing stream of a suitable diluent. The diluted sample is propelled through a small dialyser where diffusible constituents pass through the membrane and are transferred to a flowing stream of reagent, which is then continuously processed to produce a colour that is specific for the desired constituent. The coloured stream is finally passed through a flow-cell colorimeter that is equipped to record the results of the analysis. This procedure has been used for the determination of urea nitrogen and glucose in blood and of calcium, chloride, and acid and alkaline phosphatases in serum. The continuous method can be made reproducible and accurate to within 1% and results have generally been superior to those obtained by methods used in routine clinical analyses. P. NICHOLLS

996. **Determination of potassium in blood serum.** I. M. Korenman and N. I. Malkina. *Nauch. Rabot. Khim. Lab. Gor'kovsk. Nauch. Inst. Gigien. Trud. i Profbolezneĭ*, 1957, (6), 169–178; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 22,876.—Potassium is pptd. from blood serum as $K_2Na[Co(NO_2)_6]$, which is determined by titration with permanganate. It is necessary to take 0·15 ml of blood or 0·5 ml of serum. K. R. C.

997. **Modification of Camponovo's method for the determination of serum potassium.** O. A. Orsingher and A. Martin (Inst. Médico Claudio Bernard, Córdoba, Argentina). *Rev. Asoc. Bioquim. Argentina*, 1958, **23**, 85–89.—Beer's law is more closely followed if the concn. of HCl in the final soln. used in Camponovo's method (*cf. Rev. Soc. Arg. Biol.*, 1953, **29**, 210) is reduced to 0·1 ml of conc. HCl (*d*, 1·19) per 100 ml. The average results on 12 samples examined by this modified technique were 0·11% higher than those obtained with the normal method. G. H. FOXLEY

998. **Use of diphenylcarbazone for photometric micro-determinations of copper in plant materials.** L. N. Lapin and M. A. Rish. *Trudy Tadzh. Uchit. Inst.*, 1957, **4**, 71–78; *Ref. Zhur., Khim., Biol. Khim.*, 1958, (11), Abstr. No. 13,301.—The test material (0·5 to 0·6 g of fresh or 0·2 g of air-dried substance) is ashed with $H_2SO_4$ and $K_2SO_4$, neutralised with aq. $NH_3$ soln., and then 1 g of $KH_2PO_4$ is added. An aliquot of the liquid obtained is extracted with a 0·01% soln. of diphenylcarbazone in benzene and the colour of the benzene extract is measured in a photometer. The method employed enables microgram amounts of Cu to be determined in the presence of the other cations. The error is ± 3%.

K. R. C.

999. **Determination of copper and molybdenum in plants by X-ray spectrography.** V. A. Lazar and K. C. Beeson (U.S. Plant, Soil and Nutrition Lab., Agric. Res. Service, Ithaca, N.Y., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 416–419.—Copper cannot be determined satisfactorily by X-ray spectrography in dried ground plant tissue, but in determinations in ashed plant tissue the results are subject to a deviation which is not too large for routine purposes, as is shown by plots of the ratio $F/S$ *vs.* concn., where $F$ is a measure of the intensity of the Cu $K_\alpha$ line and $S$ is a measure of the intensity at an arbitrarily chosen background. A curve based on Cu concn. in plant tissue is inapplicable to determinations in seed. Molybdenum can be satisfactorily determined in tissue or ash.

A. A. ELDRIDGE

1000. **Determination of calcium in blood serum.** I. M. Korenman and N. I. Malkina. *Nauch. Rabot. Khim. Lab. Gor'kovsk. Nauch. Inst. Gigien. Trud. i Profbolezneĭ*, 1957, (6), 164–168; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 22,875.—Calcium is pptd. as oxalate, the washed ppt. is added to $H_2SO_4$ and the oxalic acid is titrated against permanganate. Calcium was determined in 0·03 ml of blood serum. K. R. C.

1001. **Simple spectrophotometric method for the determination of calcium.** P. V. Ferro and A. B. Ham (Dept. Chem., Dade Reagents, Inc., Miami, Fla.). *Amer. J. Clin. Path.*, 1957, **28** (6), 689–693.—This method is based on the pptn. of Ca from serum or water with Na chloranilate. The insoluble Ca chloranilate is washed with 50% *iso*propyl alcohol and then dissolved in EDTA (tetrasodium salt) forming a pink colour, the extinction of which is measured at 520 mμ. The semi-micro procedure described requires only 0·5 ml of serum. The use of a soln. of Na chloranilate instead of chloranilic acid reduces the time for the pptn. of Ca from 2 hr. to 30 min. The results have an accuracy and reproducibility similar to that for the method of pptn. with ammonium oxalate. The method may be applied to the determination of Ca in urine and spinal fluid. P. NICHOLLS

1002. **Determination of calcium and magnesium in plant ash by complexometric methods.** N. N. Karnaukhova and S. V. Pel'tikhin. *Nauch. Zap. Voroshilovgradsk. Sov. Inst.*, 1957, **4** (2), 58–62; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 9547.—To attain the greatest accuracy in the titration of total Ca and Mg, an addition of 5 milliequiv. of $NH_3$ per 100 ml of neutral ash soln. is recommended and, for the titration of Ca, sufficient NaOH should be added to give a pH > 12·5.

K. R. C.

1003. **Dithizone and zincon procedures for zinc in plants: separation of interfering elements by ion-exchange resins.** T. L. Yuan and J. G. A. Fiskell (Univ. Florida, Gainesville, Fla., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 424–428.—The sample (5 g) is digested and evaporated with $HClO_4$ and $HNO_3$; the residue is digested with 6 *M* HCl (20 ml) followed by dilution to 50 ml. An aliquot is passed through Dowex-1 resin, *M* HCl

is added, and the eluate is rejected. Washings (water) and the eluate from 0·01 *M* HCl are neutralised (aq. $NH_3$), and treated with citrate and dithizone in $CCl_4$. Zinc is determined spectrophotometrically at 535 mμ. Alternatively, zincon may be used. The average recovery of added Zn (20 μg) was 91·5%. A. A. ELDRIDGE

1004. **The determination of silicon in blood.** I. M. Korenman and O. M. Gulina. *Nauch. Rabot. Khim. Lab. Gor'kovsk. Nauch. Inst. Gigien. Trud. i Profbolezneĭ*, 1957, (6), 179–184; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 18,945.—A micro-determination of silicic acid in 0·1 ml of blood serum is made with Pb acetate in an acetic acid medium. Results are accurate to within 10% and the sensitivity is 1·4 μg. K. R. C.

1005. **Spectrum analysis of silicon and manganese in blood.** M. B. Alekseeva and V. I. Belozerskaya. *Gigiena i Sanitariya*, 1957, (12), 73–75; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 21,602.—The sensitivity of the spectrum analysis of Si is 0·00005 mg per 100 ml and of Mn is 0·001 mg per 100 ml. The limits of error are ±9·5%. This method is used to examine the blood of people working in a foundry and the Si and Mn contents are shown to be higher than normal. The Si content of blood increases with age even in those who do not work with silicon compounds. A similar increase was not observed in the case of Mn. K. R. C.

1006. **Determination of urea nitrogen with the diacetyl method and an automatic dialysing apparatus.** W. H. Marsh, B. Fingerhut and E. Kirsh (Dept. Path., State Univ., New York). *Amer. J. Clin. Path.*, 1957, **28** (6), 681–688.—Use is made of the automatic device described by Skeggs (*cf. Anal. Abstr.*, 1959, **6**, 995). Samples of oxalated blood, ≃ 2 ml, are drawn up in sequence from separate depressions in the sample plate. The carbamido - monoxime reaction is then used to determine the amount of dialysed urea. The entire analysis is complete in 3 min. Three hundred non-selected blood samples were simultaneously analysed by the automatic procedure and by a modification of the urease method for the determination of urea nitrogen. Slightly higher values were obtained with the diacetyl monoxime method than with the urease technique, but there is no clinical significance in the differences between the results of the two methods. Non-urea substances do not significantly affect the colour developed in the carbamido - diacetyl monoxime reaction. P. NICHOLLS

1007. **Determination of preformed urinary ammonia (nitrogen) by means of direct nesslerisation.** H. V. Connerty, A. R. Briggs and E. H. Eaton (Dept. Path., Veterans Administration Hosp., Wilmington, Del.). *Amer. J. Clin. Path.*, 1957, **28** (6), 634–638.—In this method the effect of interfering substances present in urine is first eliminated by the use of iodine when the amount of preformed ammonia is determined by direct nesslerisation. This simple procedure is both rapid and accurate, the average recovery being 100·6%. It is indicated that this method is also suitable when the urine contains large amounts of bile. P. NICHOLLS

1008. **Estimation of orthophosphates in the presence of acid-molybdate-labile phosphoric acid compounds.** J. E. Wahler and A. Wollenberger (Dtsch. Akad. der Wissenschaften, Berlin-Buch). *Biochem. Z.*, 1958, **329** (6), 508–520.—A systematic investigation of the colorimetric determination of phosphate by the molybdate reaction, with subsequent extraction of the reaction product, is reported. The primary product of the reaction, dodecamolybdophosphoric acid, is separated in the system 0·125 *N* $HClO_4$ - *iso*propyl acetate at 0° from labile phosphates and from isopolymolybdates and is measured directly in the organic phase by its light absorption at 310 mμ or after reduction by stannous chloride, added as solid or in acid soln., at 710 or 725 mμ, respectively. The method was shown to be valid for the analysis of blood and $HClO_4$ extracts of animal tissues. Determination of $10^{-9}$ to $2 \times 10^{-7}$ moles of orthophosphate may be achieved without significant interference by creatine phosphate or other phosphate compounds of similar lability. G. W. CAMBRIDGE

1009. **Nuclear-track technique for low-level plutonium in urine.** L. C. Schwendiman and J. W. Healy (Hanford Atomic Products Operation, General Electric Co., Richland, Washington). *Nucleonics*, 1958, **16** (6), 78–82.—The Pu is separated from the sample by a procedure involving evaporation to dryness, re-dissolving, co-pptn. with $LaF_3$, extraction with thenoyltrifluoroacetone and back-extraction into 8 *M* HCl. It is subsequently electro-deposited from 1 to 2 *M* KOH containing NaOCl on to a stainless-steel disc. After exposure of the disc to Kodak NTA emulsion (25 μ thick) for 168 hr. at 5° and development, the number of tracks is counted. The recovery of Pu, assuming a 50% emulsion geometry, is 87%. The coefficient of variation in 553 determinations of 0·57 disintegration per min. of Pu was ± 12%. G. J. HUNTER

1010. **Improved direct mercurimetric titration of chloride in biological fluids.** E. W. Rice (Dept. Path., Univ. Pittsburgh Sch. of Medicine, Pittsburgh, Pa.). *Amer. J. Clin. Path.*, 1957, **28** (6), 694–696.—The addition of diethyl ether to the titration medium enables the mercurimetric determination of chloride by the Schales and Schales method to be applied directly to serum, cerebrospinal fluid and urine without deproteinising. P. NICHOLLS

1011. **Determination of bromide "space" by means of a polarographic method.** H. S. Jones and M. L. Terry (Harvard Med. Sch., Boston, Mass., U.S.A.). *J. Lab. Clin. Med.*, 1958, **51** (4), 609–615.—Details are given of a polarographic method for determining $Br^-$ in body fluids at concn. of 1·0 to 10·0 milli-equiv. per litre on 0·1 ml of serum. The diffusion current, measured at − 0·230 V against a mercurous sulphate half-cell, is proportional to $Br^-$ concn. and is not affected by $Cl^-$ in the range 80 to 120 milli-equiv. per litre. W. H. C. SHAW

1012. **Determination of 4-aminosalicylic acid in biological liquids.** O. M. Gulina. *Nauch. Rabot. Khim. Lab. Gor'kovsk. Nauch. Inst. Gigien. Trud. i Profbolezneĭ*, 1957, (6), 132–139; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 17,539.—A new quant. micro-method for the determination of 4-aminosalicylic acid in pure soln. and in biological fluids with *p*-aminophenylmercury acetate is described. Results are accurate to within ≃ 2%, and the sensitivity is 1 μg in 3 to 4 ml. K. R. C.

1013. **Quantitative determination of sugar in urine by titration.** Yu. L. Kuz'mina and V. V. Dyudyaev. *Sb. Nauch. Trud. Ivanovsk. Med. Inst.*, 1957, (12), 478–482; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 22,894.—It is confirmed that Althausen's method gives very low results when the sugar content of urine is high and high results when the sugar content is low. Conditions are specified for a Fehling's soln. titration to give results easily reproducible by polarimetric methods. The results are tabulated. K. R. C.

1014. **Quantitative method for estimation of some carbohydrates on filter-paper.** J. W. Date (Inst. of Hygiene and Inst. of Biochem., Univ. of Aarhus, Denmark). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (2), 149–154.—Spots containing 15 to 1000 $\mu$g of glucose, galactose, xylose, lactose and arabinose on paper chromatograms have been sprayed with an aniline reagent containing equal parts of alcoholic aniline (1·10 *M*) and aq. citric acid (1 *M*). After the paper has been heated to 100° for 10 min. the carbohydrates are seen as brown spots. The spots and blanks of equal areas are cut out and each is eluted in 10 ml of *n*-butanol - pyridine - water (2:2:1, by vol.) over 5 hr. with regular inversion. The colour is assayed spectrophotometrically at 420 m$\mu$. Average recoveries of carbohydrate from salt-containing soln. are within 8% of the expected values. D. W. Moss

1015. **Micro-method for determination of blood galactose by means of glucose oxidase (notatin) and anthrone.** G. Søndergaard (Dept. of Medicine, Aarhus Amtssygehus, Aarhus, Denmark). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (2), 203–210.—Blood (0·1 ml in 1 ml of water) is incubated for 90 min. at 37° with 0·2 ml of a soln. of glucose oxidase at pH 5·8, thus oxidising glucose to gluconic acid. Proteins are pptd. by addition of $Ba(OH)_2$ and $ZnSO_4$, followed by centrifugation. The protein-free soln. (1 ml) is mixed with 5 ml of anthrone - $H_2SO_4$ reagent (1 g of anthrone in 500 ml of 83% $H_2SO_4$) and heated in the boiling-water bath for exactly 4 min. After cooling the soln. in ice-water and at room temp., the green colour is read at 620 m$\mu$. A galactose standard and a blank are treated similarly. Recoveries of galactose added to blood are of the order of 100%. The pH is close to the enzyme optimum, and the time and temp. conditions are adequate for the oxidation of all but excessively elevated blood-glucose concn. The enzyme preparation is stable for several months in the refrigerator. D. W. Moss

1016. **Determination of polysaccharides in human blood serum.** D. A. Tsuverkalov and V. M. Khramov. *Vrachebnoe Delo*, 1957, (9), 983–986; *Ref. Zhur., Khim., Biol. Khim.*, 1958, (11), Abstr. No. 13,230.—Venous blood ($\simeq$ 2 ml) was placed in a thermostat at 37° to 38° for 20 to 30 min. and then centrifuged. The sugar content of the serum was determined by the Hagedorn and Jensen method. Serum (0·5 ml) was heated on a water bath at 65° for 5 min. with phosphate buffer (1·15 ml) at pH 6·5; dry, powdered pancreatin (10 mg) was added and the mixture was placed in a 45° thermostat for 3 hr. together with the control (2 ml of buffer with 10 mg of pancreatin). The quantity of sugar found in the control tube was subtracted from the quantity of sugar found in the experimental tube and the remainder was multiplied by four; this gave the total quantity of sugar in serum after fermentative hydrolysis. The quantity of sugar after hydrolysis varied between 262 and 298 mg per 100 ml for normal people (10 samples). K. R. C.

1017. **Determination of lactic acid in blood.** I. M. Korenman and O. M. Gulina. *Nauch. Rabot. Khim. Lab. Gor'kovsk. Nauch. Inst. Gigien. Trud. i Profbolezneĭ*, 1957, (6), 114–123; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 22,874.—The iodimetric method described is based on a permanganate oxidation of lactic acid to acetaldehyde. Lactic acid was determined in 0·2 ml of blood with an average error of 10% and in 0·1 ml with an average error of 15%. K. R. C.

1018. **An optical - enzymic estimation of L-(+)-lactic acid using a lactic acid dehydrogenase from yeast which is independent of DPN.** O. Wieland (II Med. Klinik, Univ. München, Germany). *Biochem. Z.*, 1958, **329** (7), 568–576.—The reduction of ferricyanide by DPN-independent lactic acid dehydrogenase is the basis of a method for the determination of lactic acid in blood, cerebrospinal fluid and other fluids. The limit of detection is 1 $\mu$g in 1 ml and the mean error is 2%. Glyceric acid does not interfere with the estimation unless present in very high concn. *Procedure*—In 2 tubes are placed 0·07 *M* pyrophosphate buffer (pH 8·0) (1 ml) and 0·01 *M* $K_3Fe(CN)_6$ (0·15 ml). The appropriate amount of protein-free neutralised filtrate is added. In the control tube the volume is made up to 2 ml with water and in the other tube the enzyme is added in addition. The amount of lactic acid dehydrogenase added is 0·01 ml of a soln. equiv. to 480 $\mu$g of protein. The tubes are incubated for 10 to 15 min. at 30°. The extinction is measured at 405 m$\mu$. There is a straight-line relationship between extinction and concn. of DL-lactate over a range of 0·05 to 0·65 $\mu$moles per ml. G. W. Cambridge

1019. **Determination of pyruvic acid in blood.** O. M. Gulina. *Nauch. Rabot. Khim. Labor. Gor'kovsk. Nauch. Inst. Gigien. Trud. i Profbolezneĭ*, 1957, (6), 102–113; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 16,106.—Several colorimetric methods were investigated. Salicylic acid and 2:4-dinitrophenylhydrazine were tested as reagents, and known volumetric methods for the determination of pyruvic acid in 2 ml of blood were examined. The pyruvic acid content varied according to the method used. A modified volumetric method was produced for 2 ml of blood, and this gave better results, enabling the pyruvic acid to be determined in 0·1 ml of blood with a relative error of $\pm$ 16% and a sensitivity of 0·95 $\mu$g (0·1 ml). K. R. C.

1020. **Chromatographic methods for the determination of organic acids in plants.** A. E. Petrov-Spiridonov. *Izv. Timiryazevsk. Soviet Akad.*, 1957, (2), 230–234; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 8269.—Methods for the estimation of organic acids on a silica-gel column and by means of paper chromatography are described. The experimental error in tests with pure organic acids on the silica-gel column were $\pm$ 10% for 1 to 3 mg of acid and $\pm$ 0·5% for 10 to 16 mg. A mixture of *n*-butanol and $CHCl_3$ was used as solvent. The beginning and end of the test were shown by a change in the colour of the thymol blue indicator. The amount of acid was determined by titration against 0·01 *N* $Ba(OH)_2$ with the same indicator. The separation of the organic acid mixture requires

2 to 3·5 hr. An *n*-butanol - acetic acid - water mixture (3:1:2) or a *n*-butanol - formic acid - water mixture (4:1:2) was used as solvent in paper chromatography. An ether - formic acid - water mixture (10:7:1) was used for quant. determinations. A 0·04% soln. of bromophenol blue in 95% ethanol was used for colour development.

K. R. C.

1021. **Quantitative determination of urea in blood.** B. A. Rashkovan. *Sb. Nauch. Rabot Vitebskiĭ Med. Inst.*, 1957, (8), 108–118; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 17,512.—Blood (0·1 to 0·2 ml) is mixed with ethanol (2 ml), set aside for 5 to 10 min., and filtered through paper washed with ethanol into a graduated test-tube. The filter-paper is washed with ethanol (× 3), the filtrate is made up to 7 ml with ethanol, then $H_2O$ (2 ml) and 0·035 *N* HCl (1 ml) are added. After shaking the mixture, a 1·2% soln. of hypochlorite (1 ml) is added, followed, after further shaking, by a 5% soln. of phenol (1 ml). The test-tube is heated on a water bath at 55° for 25 min., then the soln. is measured colorimetrically against a standard prepared from soln. of pure urea of corresponding concn. K. R. C.

1022. **Determination of vitamin E in blood serum.** A. M. Épel'baum and G. M. Lushchevskaya. *Vitaminy*, 1958, (3), 95–100; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 22,873.—A simplified method, suitable for clinical purposes, involves deproteinisation by ethanol, extraction with benzene, removal of extraneous substances from the benzene extract by means of chromatographic adsorption on diatomite, and finally the quant. determination of vitamin E by the iron - 2:2′-dipyridyl method. K. R. C.

1023. **Determination of bilirubin in blood serum.** I. M. Korenman and F. S. Frum. *Nauch. Rabot. Khim. Lab. Gor'kovsk. Nauch. Inst. Gigien. Trud. i Profbolezneĭ*, 1957, (6), 140–144; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 22,871.—Ethanol (96%) is added to blood serum, the mixture is centrifuged, and a freshly prepared soln. of diazo reagent (0·5 ml) and 96% ethanol (1 ml) is added to the centrifugate. A pink colour appears after 5 to 10 min. and this is determined colorimetrically. K. R. C.

1024. **Photometric method for the determination of bilirubin in horse serum.** B. V. Sidorenko. *Sb. Trud. Leningr. Nauch. Inst.*, 1957, (7), 213–217; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 17,074.—A photometric method is described for the determination of bilirubin in blood serum with the aid of caffeine and a diazo reagent. K. R. C.

1025. **Determination of porphobilinogen, copro- and uro-porphyrin in urine and tissue. Preliminary report.** T. Heikel (Dept. of Med. Chem., Univ. of Helsinki, Finland). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (2), 193–195.—Urine, diluted if necessary, or homogenised tissue is added to Dowex-2 anion-exchange resin. Porphobilinogen is displaced with acetic acid (50% soln.) and is determined spectrophotometrically with Ehrlich reagent at 555 m$\mu$. Coproporphyrin is determined in the same eluate by making alkaline to Congo red with satd. Na acetate soln., extracting into ether and then into HCl (0·1 *N*) and measuring the extinction at 402, 380 and 380 m$\mu$. The dry resin, free from acetic acid, is treated with methanol saturated with HCl vapour, eluting uroporphyrin. On setting aside the eluate for 24 hr., esterification takes place. The esters are extracted first with $CHCl_3$, then with HCl (7 *N*), and uroporphyrin is determined spectrophotometrically at 410, 430 and 380 m$\mu$ after 4 days. Recoveries of 80 to 95% for porphobilinogen, 90 to 95% for coproporphyrin and 70 to 80% for uroporphyrin, with the pure substances, are obtained. D. W. Moss

1026. **A simplified Lucite cuvette for the spectrophotometric measurement of haemoglobin and oxyhaemoglobin.** G. G. Nahas (Dept. of Cardio-respiratory Diseases, Walter Reed Army Med. Center, Washington, D.C.). *J. Appl. Physiol.*, 1958, **13** (1), 147–152.—Use of the simply constructed cuvette described with the Beckman DU spectrophotometer enables haemoglobin and oxyhaemoglobin to be determined by measurements of extinction at 660 and 805 m$\mu$. This has the advantage that a relatively thick (0·1 cm) layer of undiluted blood can be used, and it is not necessary to prevent exposure to air above the light-path if readings are made within 10 min.

D. W. Moss

1027. **Quantitative spectrophotometric determination of haemoglobin by Soret's absorption band.** T. N. Gladyshevskaya, Ya. S. Kvyatkovskaya and B. A. Sobchuk. *Ukr. Biokhim. Zhur.*, 1957, **29**, 371–374; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 10,852.—It has been shown that, with dilutions of blood from 1:500 to 1:1500, haemoglobin can be determined by measurements of absorption at 417 m$\mu$ (Soret's method). Simultaneous determinations of haemoglobin by a gasometric method demonstrated the applicability of the Beer - Lambert law. K. R. C.

1028. **Separation of chlorophylls by paper and cellulose-powder column chromatography.** A. Angapindu, H. Silberman and P. Tantivatana (Sydney Univ., Australia). *Arch. Biochem. Biophys.*, 1958, **75** (1), 56–68.—This survey includes an examination of adsorption methods on filter-paper strips and on paper impregnated with sucrose, $CaCO_3$ or $ZnCO_3$ and developed by horizontal or vertical techniques. Procedures based predominantly on partition, including reversed-phase methods, on both paper strips and circular papers are also investigated and the value of cellulose powder for the separations is discussed. From a study of the conditions required for the separation of the different chlorophylls and for their quant. recovery in a spectroscopically pure state, two techniques are recommended—(*i*) reversed-phase chromatography on Whatman No. 4 paper impregnated with liquid paraffin and irrigated with methanol, and the use of either circular papers or paper strips, (*ii*) circular chromatography on unimpregnated paper. By a combination of these two techniques, separation of the natural chlorophylls and some carotenoids is achieved. Precautions necessary to avoid losses during development and elution are discussed. W. H. C. Shaw

1029. **The structure and identification of hair.** R. J. Myers (Colgate Univ., Hamilton, N.Y., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 368–376.—Hairs may be classified in sub-divisions of the two main groups according to the presence or absence of erectile tissue in the follicles. The microscopical characteristics of the cuticle, cortex,

medulla and pigment are described. Details of microscopical techniques employed in ascertaining the type of donor or location of the source of the hair are reviewed. A. A. ELDRIDGE

1030. **High-voltage paper electrophoresis. A rapid method for determination of urinary amino acids.** J. K. Visakorpi and A.-L. Puranen (Children's Clinic, Univ. of Helsinki, Finland). *Scand. J. Clin. Lab. Invest.*, 1958, **10** (2), 196–202.—Dialysed urine (10 to 100 $\mu$l) is submitted to electrophoresis on paper at pH 2·25 in a buffer containing equal vol. of formic acid (0·75 *N*) and acetic acid (1 *N*) for 150 min. A voltage of 55 V per cm length and a current of 1·0 to 1·1 mA per cm width are employed, with refrigeration at — 13° to — 15°. The spots are located with ninhydrin, and the method can be made quant. by eluting the coloured areas into $CuSO_4.5H_2O$ soln. (0·2% in 75% ethanol) and reading the extinction at 520 m$\mu$ (400 m$\mu$ for histidine) after 30 min. A glutamine standard is used. The paper has a high colour value which is compensated for by the elution of blank areas. Separation of amino acids is generally good. Fairly good correlation was observed in a series of 10 urines from healthy children between the amount of amino-nitrogen determined electrophoretically and that found by a method using Folin's naphthaquinone reaction. D. W. Moss

1031. **The use of turbidity measurements in the microbiological determinations of amino acids. I.** S. S. Schiaffino, J. J. McGuire and H. W. Loy (Food and Drug Admin., U.S. Dept. of Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 420–423.—Williams' procedure (*Mem. Cornell Univ. Agric. Exp. Sta.*, 337, Jan. 1955), with modifications, was used to determine turbidity values after incubation at 34° ± 0·5° for 18 hr. The method was applied to the following amino acids by means of the organisms indicated—*iso*leucine, leucine, tryptophan, valine (*Lactobacillus plantarum, Streptococcus faecalis*), lysine (*Leuconostoc mesenteroides*), methionine, phenylalanine (*Lb. plantarum, Ln. mesenteroides*), threonine (*Strep. faecalis*). Response curves are reproduced. Satisfactory results were obtained. Advantages over the titrimetric method are increased sensitivity, shortened incubation time, easier measurement of response, and more rapid calculation of results with the same degree of accuracy. A. A. ELDRIDGE

1032. **Separation of basic amino acids with two-dimensional paper electrophoresis.** L. Rexová-Berková and O. Markovič (Dept. Pharm. Chem. and Biochem., Chem. Inst. Acad. Sci., Bratislava, Czechoslovakia). *Chem. Listy*, 1958, **52** (5), 978–979.—The mixture of amino acids is first separated by the use of descending paper electrophoresis into neutral, acid and basic fractions, and the basic fraction is further separated by horizontal electrophoresis in a basic electrolyte. For the first separation a pyridine - acetate buffer soln. (pH 5·6) is used, and for the final separation a borate (pH 11·2) or 0·05 *N* NaOH electrolyte is suitable. The detection is carried out with a 0·5% acetone soln. of ninhydrin after previous neutralisation of the paper with 1% acetic acid. J. ZÝKA

1033. **An electrophoretic method for the separation of amino acids from sugars. (Preliminary report.)** Yu. Novitskiĭ. *Fiziol. Rasteniĭ, Akad. Nauk SSSR*, 1957, **4** (1), 103–105; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 8197.—A method is described for the separation of simple sugars from amino acids in plant leaves by means of paper electrophoresis at 6000 V. Electrophoresis was continued until the proteins, cellulose, starch, organic acids and other substances were removed from the leaf extracts. It was found that after electrophoresis for about 20 to 25 min. under the given conditions a complete separation of sugars from amino acids was effected. K. R. C.

1034. **Application of high-tension electrophoresis to the total quantitative analysis of protein hydrolysates. II.** K. Dose (Max Planck Inst. f. Biophysik, Frankfurt a.M., Germany). *Biochem. Z.*, 1958, **329** (5), 416–420.—A scheme for the separation of amino acids from protein hydrolysates is described and its application to lysozyme and to serum albumin is illustrated. Details of most of the techniques used have been reported (*cf.* Dose and Caputo, *Biochem. Z.*, 1956, **328**, 376). The outline of the scheme is as follows. Electrophoresis at 35 V per cm for 90 min. at pH 6·0 enables lysine, arginine, histidine, glutamine and asparagine to be separated from neutral amino acids which, after elution, are subjected to electrophoresis at 50 V per cm at pH 2·0 for 70 min. By this means glycine, alanine, tyrosine and tryptophan are separated from two fractions which have different mobilities. After elution these two fractions are subjected to chromatography in *sec.*-butyl alcohol - formic acid - water (15:3:2) for 18 hr. These two fractions yield (*a*) leucine and *iso*leucine, valine, methionine, proline, serine and threonine, and (*b*) phenylalanine and cysteine, respectively. Details of the techniques, including the photometric estimation of the amino acids, are briefly reported. G. W. CAMBRIDGE

1035. **Determination of $\gamma$-aminobutyric acid and glutamic acid in biological material by high-voltage electrophoresis.** A. Hanson and W. von Studnitz (Chem. Zentrallab. d. Allg. Krankenhauses, Malmö, Sweden). *Acta Chem. Scand.*, 1958, **12** (6), 1332–1333 (in German).—Glutamic acid and $\gamma$-aminobutyric acid may be rapidly determined simultaneously and with reasonable accuracy by subjecting an alcoholic extract of the material to electrophoresis at 55 V per cm on Macherey & Nagel Paper No. 214 in a buffer medium of conc. formic acid - glacial acetic acid - water (3:2:15, by vol.) for 75 min. The paper is dried at 75° for 0·5 hr. in a drying cabinet and then sprayed with a 0·5% ninhydrin soln. in *n*-butanol which is saturated with pyridine - glacial acetic acid - water (10:1:95) (pH 6). After drying the electropherogram for 20 min. near a fan at about 40° and heating at 65° for 20 min. in a cabinet, the coloured flecks are converted into the corresponding red copper complexes. These flecks are then extracted with methanol and the light absorption of the extract is measured in a Beckman spectrophotometer at 504 m$\mu$. Glutamic acid moves 10·5 cm and $\gamma$-aminobutyric acid moves 15·8 cm towards the cathode under the electrophoresis conditions employed. If large amounts of glycine are present in the material, a buffer medium of glacial acetic acid - pyridine - water (15:1:89, by vol.) (pH 3·5) should be used in order to avoid an "overloading" effect on $\gamma$-aminobutyric acid. S. M. MARSH

1036. **Determination of glutathione in blood.** I. M. Korenman and N. I. Malkina. *Nauch. Rabot. Khim. Lab. Gor'kovsk. Nauch. Inst. Gigien.*

*Trud. i Profbolezneĭ*, 1957, (6), 145–153; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 22,872.—The quantity of reduced glutathione in a portion of protein-free blood filtrate is determined iodimetrically. The oxidised glutathione (hexapeptide) in another portion is reduced and determined quant.; the amount of oxidised glutathione is deduced. The glutathione content of 0·03 to 0·1 ml of blood is determined. K. R. C.

1037. **Methods for the extraction of proteins from brain tissue before separating them by paper electrophoresis.** N. M. Polyakova and O. P. Gotovtseva. *Ukr. Biokhim. Zhur.*, 1957, **29** (4), 400–408; *Ref. Zhur., Khim., Biol. Khim.*, 1958, (17), Abstr. No. 21,551.—After washing cat-brain free of blood with physiological saline, the membranes are removed and the tissue is pulverised in a mortar. The paste is extracted for 2 hr. in a refrigerator in the same or in a double vol. of liquid and twice centrifuged (20 and 10 min.) at 8000 r.p.m. The proteins and protein nitrogen in the soln. and in the ppt. are determined quant. by means of the biuret reagent and by nitrogen estimations, respectively. A study was made of each of the following extractive media —succinic acid buffer soln. at pH 3·6 and 4·2, acetate buffers at pH 5·0 and 5·6, $H_2O$, physiological saline at pH 6·5, physiological saline with a subsequent freezing in liquid air, a 1·16% soln. of KCl, a barbitone sodium soln. at pH 8·6, and a borate buffer soln. at pH 9·25. Percentages of proteins extracted from the brain were 3·9, 5·2, 8·3, 12·8, 15·2, 17·0, 16·6, 17·5, 20·7 and 20·3, respectively. The extracts obtained by the use of barbitone sodium and borate buffer soln. were turbid and viscous, possibly owing to the extraction of lipids. The clearest soln. and the best separations of proteins by paper electrophoresis were obtained by the use of physiological saline and subsequent freezing in liquid air. A similar extraction of lyophilised brain did not give such a well-defined electropherogram. K. R. C.

1038. **Stabilised tungstic acid reagent for blood deproteinisation.** W. T. Caraway (Flint Med. Lab., Flint, Michigan, U.S.A.). *Chemist Analyst*, 1958, **47** (2), 44–45.—A reagent that is stable for at least four months is made in two concn.—(*i*) for whole blood mix, in order, water (700 ml), sodium tungstate dihydrate soln. (100 ml of 10%), $H_3PO_4$ (0·1 ml of 85%) and $H_2SO_4$ (100 ml of 0·67 *N*); (*ii*) for serum or plasma, as (*i*), but with 1600 ml of water. For use, add rapidly 9 vol. of reagent to 1 vol. of blood, mix, allow to stand for 5 min., then centrifuge or filter. G. S. Roberts

1039. **The bound carbohydrates of isolated plasma proteins.** H. E. Schultze, R. Schmidtberger and H. Haupt (Behringwerke, Marburg a.d. Lahn). *Biochem. Z.*, 1958, **329**, 490–507.—Protein-bound galactose, mannose, fucose, hexosamine, neuraminic acid and hexuronic acid in plasma were determined colorimetrically. Methods for improving the differentiation between galactose and mannose, as well as for determining neuraminic and uronic acids, are given. The interference of accompanying sugars in the specific colour development is eliminated by the use of several standard curves with graded amounts of the disturbing components.

Nutr. Abstr. Rev.

1040. **Paper electrophoresis of blood-serum proteins.** V. N. Dzyak and A. M. Bulȳkh. *Sb. Nauch. Rabot Dnepropetr. Med. Inst.*, 1956, (1), 357–363; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 9506.—A detailed method is described. A densitometer, constructed by the authors, is recommended for the quant. determination of the electropherogram. K. R. C.

1041. **Separation of blood-serum proteins by filter-paper electrophoresis.** A. G. Malakhov. *Trudy Vses. Inst. Eksp. Veterinarii*, 1957, **20**, 312–320; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 17,470.—The method is described in detail. Best results were obtained with a buffer soln. consisting of barbitone sodium (5·16 g), barbitone (0·9 g), and $Na_2HPO_4.2H_2O$ (5 g) in 1 litre of soln. Well-defined electropherograms were obtained with this buffer soln. and the usual filter-paper, at 230 V and 0·5 to 0·7 mA per cm for 18 hr. Quant. determinations were carried out, after elution, with the aid of planimetric curves. K. R. C.

1042. **Electrophoretic investigation of soluble liver proteins by optical electrophoresis.** I. I. Rodinov. *Trudy Krym. Med. Inst.*, 1957, **17**, 63–69; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 16,100.—The soluble liver proteins of rabbits, dogs, sheep, bulls and lions were investigated. The liver was washed free of blood with physiological saline, cut up into small pieces and twice frozen and thawed out. The material was cut up into fine sections, which were extracted with a soln. of salts for 120 hr. at 2°. An extraction of liver with a 1·0 *M* KCl soln., pH 7·0, $\mu$ 1·0, and with a mixture of 1·0 *M* KCl and 0·03 *M* $NaHCO_3$, pH 8·5, $\mu$ 1·0, was found to be best for the electrophoresis of protein soln. By this method, the protein content of the extract was 1·2 to 2%. The extract was dialysed at 2° against a barbitone buffer, pH 8·6, $\mu$ 0·1. Electrophoresis was carried out in an apparatus which was operated according to the Troitsky method—the buffer soln. was maintained at 4° and the potential gradient at 4 to 5 V per cm for 1·5 to 2 hr. Six fractions were defined quant. and the specific differences in the relative proportions of these fractions were noted. K. R. C.

1043. **The suitability of Light green as a stain for the quantitative evaluation of proteins in paper electrophoresis.** V. Bonavita and V. Scardi (Inst. of Human Physiology, Univ. of Naples, Italy). *J. Chromatography*, 1958, **1** (3), 285–287.—Light green is not suitable for the quant. determination of protein in paper electrophoresis because of non-proportionality between dye uptake and protein concn. A. R. Rogers

1044. **Nigrosine as a stain for quantitative estimation of serum proteins in paper electrophoresis.** V. Scardi and V. Bonavita (Inst. of Human Physiology, Univ. of Naples, Italy). *J. Chromatography*, 1958, **1** (3), 287–289.—Nigrosine is not suitable for the quant. determination of protein in paper electrophoresis because the dye uptake varies with protein concn., as well as from one protein to another, and because it stains the paper.

A. R. Rogers

1045. **Quantitative determination of protein fractions in blood serum by photometric measurement of paper electropherograms in reflected light.** N. Zorkina. *Trudy Stalinabadsk. Med. Inst.*, 1957, **27**, 69–71; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr.

No. 27,050.—Results obtained from the quant. determination of protein fractions separated by paper electrophoresis, and then measured photometrically in reflected light, are compared with data from a photometric analysis of individual strips.
K. R. C.

1046. **Fractionation of protein in blood serum by the salts of heavy metals.** P. P. Tolok. *Doklady Akad. Nauk UzSSR*, 1957, (10), 73–75; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 18,885.—The serum was diluted with a barbitone sodium buffer of pH 9·64 and then fractionated by $CdSO_4$. The quantity of pptd. protein was determined by a thermophoto-electrometer against a casein standard. The results thus obtained were compared with those obtained by refractometric determinations and by paper electrophoresis. The concn. of $CdSO_4$ required for the pptn. of $\gamma$-globulin was 0·008%, for $\beta$-globulin 0·0185%, for $\alpha_1$-globulin 0·022%, for $\alpha_2$-globulin 0·023% and for albumin 0·128%.
K. R. C.

1047. **Determination of the structure of protein substances with the aid of fluorodinitrobenzene.** N. N. Mel'tova and N. I. Polotnova. *Sb. Nauch. Rabot Leningr. Inst. Sov. Torgovli*, 1957, (11), 124–133; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 20,260.—The Sanger method is used to determine the N-terminal amino acids of wheat gliadin. Fluorodinitrogliadin is hydrolysed for 6 hr. by 6 *N* HCl, and the fluorodinitro derivatives of the amino acids are extracted with ether and separated by two-dimensional paper chromatography with phenol - $H_2O$ and phenol - cresol - $H_2O$. It is shown that the N-terminal amino acids of gliadin are aspartic acid and threonine. K. R. C.

1048. **Paper electrophoresis of lipo- and glyco-proteins in blood serum.** I. Todorov and A. Karakashev. *S'vrem. Med.*, 1956, **7** (6), 84–88; *Ref. Zhur., Khim., Biol. Khim.*, 1958, Abstr. No. 8202.—A method is described for the electrophoretic separation of lipo- and glyco-proteins of blood serum with various solvents and dyes. In comparison with Sudan black B, much better results were obtained with the dye, oil red 7B. Glycoproteins showed up well with Schiff's reagent. A method is described for studying the lipo- and glycoprotein fractions of serum in nephrosis. K. R. C.

1049. **Method for determination of albumin in fluids, particularly urine.** Miles Laboratories Inc. Brit. Pat. 799,626; date appl. 5.11.56.—The active ingredients of the tablet described, on which a test portion of the test liquid is to be deposited, are defined as a protein precipitant, a dye adapted to be absorbed by the pptd. protein and show a colour change at a pH $\gtrsim$6, and an effervescent couple for releasing the active ingredients.
H. F. W. Kirkpatrick

1050. **Hydrolysis of $^{131}$I-thyroprotein by pancreatic enzymes. [Determination of $^{131}$I-containing compounds in labelled thyroid tissue.]** W. Tong and I. L. Chaikoff (Dept. of Physiol., Univ. of California Sch. of Med., Berkeley, U.S.A.). *J. Biol. Chem.*, 1958, **232** (2), 939–950.—An enzymic hydrolysis technique is used which gives $\simeq$ 95% recovery of the $^{131}$I-thyroglobulin in the form of $^{131}$I-amino acids. All of the $^{131}$I is accounted for on a single paper chromatogram of a pancreatin hydrolysate. The $^{131}$I is determined by a standard method, and the alkaline hydrolysis and tedious butanol extraction and solvent evaporation stages of older methods are eliminated. During enzymic digestion, max. release of mono-iodotyrosine occurs within 1 hr., and release of di-iodotyrosine requires $<$10 hr., whereas max. release of thryoxine needs 8 to 24 hr. J. N. Ashley

1051. **Detection of pyrimidine deoxyribosides and deoxyribotides on paper chromatograms.** J. Jonsen, L. Haavaldsen and S. Laland (Univ. of Oslo, Blindern, Norway). *J. Chromatography*, 1958, **1** (3), 291.—Dab the dried chromatogram with cotton wool soaked in Br - $H_2O$ - acetic acid (1 : 50 : 10, by vol.), dry at 100° and treat with diphenylamine - $H_2SO_4$ reagent (*cf.* Buchanan *et al.*, *J. Chem. Soc.*, 1950, 3162). Thymidine, deoxycytidine, deoxyuridine, thymidylic acid and deoxycytidylic acid all give blue spots. Bromination increases the sensitivity of the diphenylamine reagent about fourfold.
A. R. Rogers

1052. **Qualitative research on tissue ribonucleic acids. III. New method of separation, determination and study of the radioactivity of nucleotides without previous purification of the ribonucleic acid.** M. Ledig, J. D. Weill and P. Mandel (Inst. de Biol., Fac. de Med., Strasbourg). *Bull. Soc. Chim. Biol.*, 1958, **40** (4), 599–607.—Lipids and acid-soluble material are removed and the residue is hydrolysed with 0·5 *N* KOH (15 hr. at 37°). Treatment with $HClO_4$ removes proteins, and excess of $HClO_4$ is removed by pptn. as $KClO_4$ at 0°. The distribution and specific activity of each nucleotide can be determined by paper chromatography; alternatively, the total ribonucleic acid activity is determined by adsorption on charcoal followed by chromatography. H. F. W. Kirkpatrick

1053. **Spectrophotometric procedure for the analysis of nucleic acid purines and pyrimidines and its application to fish-sperm nucleic acids.** C. F. Emanuel and I. L. Chaikoff (California Univ. Med. Sch., Berkeley, U.S.A.). *Biochim. Biophys. Acta*, 1958, **28** (3), 550–561.—In the method described, deoxyribonucleic acid is hydrolysed with 10 *N* $HClO_4$, water is added and the solution centrifuged. The extinctions are measured at 262 m$\mu$ and at 280 m$\mu$ on a dilution of the supernatant liquid to give the total (purine plus pyrimidine) extinction. A further aliquot is treated with $AgClO_4$ and NaOH to bring the pH to between 2·0 and 2·7, when silver - purine is pptd. After centrifuging the mixture, the extinctions of the supernatant liquid are again measured and the two compounds are calculated by a two-component differential method. The recovery is 98 to 100%.
W. H. C. Shaw

1054. **Blue tetrazolium as a reagent for reducing steroids.** I. E. Bush and M. M. Gale (Dept. of the Regius Professor of Med., The Radcliffe Infirmary, Oxford, England). *Analyst*, 1958, **83**, 532–536.—Commercial samples of blue tetrazolium give rise to various amounts of pigments with reddish or purple hues, thus preventing the accurate determination of reducing steroids with this reagent on paper chromatograms. It is now shown that the reddish products are not partial reduction products but formazans derived from tetrazolium salts other than blue tetrazolium. When the method of Rutenberg *et al.* (*Cancer Res.*, 1950, **10**, 113) for the synthesis of blue tetrazolium was followed exactly, a satisfactory sample was easily obtained and it was found that the prescribed exhaustive

washing of the formazan before its oxidation is essential. The final product is also greatly improved by several crystallisations from a mixture of ethyl acetate and methanol (1 + 1). A. O. Jones

1055. **Determination of urinary 17-oxogenic steroids.** M. Chekherdemian, A. E. Arguelles and A. Ricca (Inst. Nac. de Med. Aeronáutica). *Rev. Asoc. Bioquim. Argentina*, 1958, **23**, 95–100.—The method of Norymberski *et al.* (*Lancet*, 1953, **ii**, 1276) has been modified by using dichloroethane instead of $CHCl_3$. The purity of the reagents affects the reliability of the results.

G. H. Foxley

1056. **Comparative study of the 17-oxogenic (Norymberski), Glenn - Nelson and Reddy methods for the determination of $C_{21}$ urinary steroids.** O. J. Golub, C. Sobel and R. J. Henry (Bio-Science Res. Foundation, Los Angeles, Calif., U.S.A.). *J. Clin. Endocrin. & Metabolism*, 1958, **18** (5), 522–530.—Good correlation exists between the results given by the Norymberski and Glenn - Nelson methods and clinical findings, but serious discrepancies were observed with the Reddy method. In the Glenn - Nelson method a significant proportion of steroid is left unextracted in the residual urine. Some advantages of the 17-oxogenic steroids method are indicated. H. F. W. Kirkpatrick

1057. **Chromatographic procedure for determination of urinary corticosteroids and $C_{19}$ steroids.** H. Wilson, J. J. Borris and M. M. Garrison (Nat. Inst. of Arthritis and Metab. Dis., Bethesda, Md., U.S.A.). *J. Clin. Endocrin. & Metabolism*, 1958, **18** (6), 643–661.—A comprehensive routine procedure is described. The column consists of synthetic aluminium silicate with 50% ethanol as the stationary phase. Development is made with hexane containing increasing proportions of $CHCl_3$. Three fractions are collected and two or more assays specific for particular groups are carried out on each fraction. H. F. W. Kirkpatrick

1058. **A portable micro-method for the determination of esterase activity in blood serum.** Kaichiro Kuroda, Masaaki Fujino and Koichi Irino (Dept. of Biochem. Sch. of Medicine, Tokushima Univ.). *Tokushima J. Exp. Med.*, 1957, **4** (3), 83–93 (in English).—The procedure is based on the method of Huggins and Lapides (*J. Biol. Chem.*, 1947, **170**, 467) and consists in the spectrophotometric determination, at 400 mμ, of the *p*-nitrophenol (**I**) liberated by the action of blood esterase on *p*-nitrophenyl acetate (**II**). To permit the assay of several samples obtained outside the laboratory, the esterase activity was arrested by the addition of ethanol (99%) and the samples were stored at − 14°. The substrate **II** was used in a concn. of 0·333 μmole per ml, adjusted before each series of determinations by reference to a standard curve obtained by means of soln. of **I** of various concn. in phosphate buffer medium at pH 7. The procedure for obtaining the standard curve needed for calculating blood esterase is described. By using capillary pipettes and glass tubes for the collection and storage of samples, only 50 to 100 mg of blood serum was needed for each determination, with a relative error of 3%. A. Tessler

1059. **Quantitative histochemistry of the brain. V. [Determination of the] enzymes of glucose metabolism.** M. V. Buell, O. H. Lowry, N. R. Roberts, Mei-Ling W. Chang and J. I. Kapphahn (Dept. of Pharmacol., Washington Univ. Sch. of Med., St. Louis, Mo., U.S.A.). *J. Biol. Chem.*, 1958, **232** (2), 979–993.—New micro-methods are described for measuring hexokinase, phosphoglucoisomerase, phosphofructokinase, glycogen phosphorylase, and phosphoglucomutase in 1 to 2 μg of tissue (dry wt.). The method for the isomerase depends on measurement of the fructose 6-phosphate formed in the reaction. For measurement of phosphoglucomutase, conditions are described for the hydrolysis of glucose 1-phosphate and a single-stage determination of the liberated inorganic phosphate (by means of molybdate and ascorbic acid). For the determination of phosphorylase activity, improved conditions are described for the measurement of inorganic phosphate in the presence of glucose 1-phosphate. Phosphofructokinase activity is determined by adding aldolase to the reaction mixture so as to convert hexose diphosphate, as fast as it is formed, into triose phosphates, which are trapped by hydrazine and are then determined colorimetrically with 2:4-dinitrophenylhydrazine. The determination of hexokinase is based on the conversion of glucose 6-phosphate, formed by the enzyme, into triose phosphate by the addition of phosphofructokinase, phosphoglucoisomerase, and aldolase. The triose phosphate is determined by the above method.

J. N. Ashley

1060. **Method for the colorimetric determination of α-D-glucosidase with a chromogenic substrate.** J. A. Goldbarg, K.-C. Tsou, S. H. Rutenburg, A. M. Rutenburg and A. M. Seligman (Harvard Med. Sch., Boston, Mass., U.S.A.). *Arch. Biochem. Biophys.*, 1958, **75** (2), 435–442.—The method is based on the liberation of 6-bromo-2-naphthol from 6-bromo-2-naphthyl α-D-glucopyranoside as substrate by α-D-glucosidase. The free naphthol is then determined by reaction with tetrazotised *o*-dianisidine to form an azo dye, which is extracted with $CHCl_3$ and determined colorimetrically. Details for preparing the substrate are given. The method is applied to the assay of serum and of rat and dog tissues; results for various organs are given and these are discussed. W. H. C. Shaw

1061. **Assay of tissue phosphoglucomutase activity.** O. Bodansky (Div. of Metabol. and Enzyme Studies, Sloan-Kettering Inst. for Cancer Res., New York, N.Y., U.S.A.). *J. Biol. Chem.*, 1958, **232** (2), 859–866.—The activity is measured by the conversion of the acid-labile P of glucose 1-phosphate into the acid-stable P of glucose 6-phosphate in accordance with well-known methods. The determination is carried out in tissue homogenates at 37°, at optimum pH 7·5 to 7·6, with optimum concn. of $1·2 \times 10^{-3}$ *M* added $Mg^{2+}$, $8 \times 10^{-3}$ *M* added L-cysteine, and at a calculated infinite concn. of the co-enzyme, α-glucose 1:6-diphosphate. J. N. Ashley

1062. **Rapid micro-method for the determination of alkaline serum phosphatases.** P. Kesselman. *Rev. Asoc. Bioquim. Argentina*, 1958, **23**, 90–94.—The serum is treated with disodium phenyl phosphate in an alkaline buffer soln. (pH 10) at 37° for 15 min. Proteins are then pptd. with trichloroacetic acid, the soln. is centrifuged and the supernatant liquor is made alkaline and treated with diazotised *p*-nitroaniline. The extinction is measured within 15 min. with a No. 47 filter.

G. H. Foxley

1063. **Micro-method for the determination of acid serum phosphatases.** P. Kesselman. *Rev. Asoc. Bioquim. Argentina*, 1958, **23**, 94–95.—The method is similar to that used for the determination of alkaline serum phosphatases (*cf. Anal. Abstr.*, 1959, **6**, 1062), except that the serum is treated with disodium phenyl phosphate in an acid buffer soln. (pH 4·9 to 5·0) at 37° for 1 hr. G. H. FOXLEY

1064. **Direct spectrophotometric determination of chlorogenic acid oxidase activity.** E. C. Sisler and H. J. Evans (N. Carolina State Coll., Raleigh, U.S.A.). *Biochim. Biophys. Acta*, 1958, **28** (3), 638–639.—Enzyme activity is determined from the change in extinction measured in a 1-cm cell at 326 mμ of $5{\cdot}7 \times 10^{-5}$ *M* chlorogenic acid in 0·1 *M* potassium phosphate buffer (pH 7·0) with $10^{-3}$ *M* EDTA. The extinction is observed at 15-sec. intervals for 75 sec. after the addition of enzyme, sufficient of which should be added to cause a decrease of 0·020 in extinction per 15 sec. The result is expressed as units, defined as the amount of enzyme that brings about an extinction change of 0·001 in the interval between 15 and 75 sec. after enzyme addition. W. H. C. SHAW

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*See also Abstracts*—**921**, Detection of Pu in wounds. **922**, Determination of Pu in humans. **960**, Determination of carbohydrates. **962**, **963**, Determination of lactic acid. **965**, Chromatographic separation of aliphatic amines. **966**, Titration of cysteine. **970**, U.v. spectra of hydroxycinnamic acids. **1069**, Determination of reserpine in plasma. **1096**, Determination of 6 sugars in honey.

## Pharmaceutical analysis

1065. **A review of conductimetric and high-frequency titrimetry in non-aqueous solutions. Implications in pharmaceutical analysis.** W. C. Purdy and J. T. Stock (Dept. of Chem., Univ. of Connecticut, Storrs). *Drug Standards*, 1958, **26** (5), 177–181.—A review. (74 references.)
A. R. ROGERS

1066. **New method of identifying opium alkaloids by paper chromatography.** J. Massicot (Lab. Chim. Biol., Fac. des Sci., Lyons, France). *Compt. Rend.*, 1958, **246** (10), 1622–1624.—As little as 0·05 μg each of morphine, codeine, thebaine, narcotine and papaverine can be separated from opium and determined semi-quant. by the micro-chromatographic procedure described. The chromatogram is run on Whatman No. 1 paper, buffered to pH 9 (NaOH - $H_3BO_3$), with diethyl ether - dioxan - acetic acid - water (13:7:2:2) as solvent mixture. The reddish spots (with yellow border) are revealed with bismuth potassium iodide (Munier and Macheboeuf, *Bull. Soc. Chim. Biol.*, 1949, **31**, 1144) formed on the paper by spraying with soln. of 0·07% aq. KI and 0·1% $BiO.NO_3$ in aq. 4% $HClO_4$. Of other alkaloids tested, only atropine and brucine gave spots of the same coloration. By using unbuffered paper and 0·16% KI with 0·1% $BiO.NO_3$ in 0·5% $HClO_4$, it is possible to detect 0·005 μg of morphine or codeine. This colour reaction is probably specific for the piperidine nucleus.
W. J. BAKER

1067. **Chromatographic - spectrophotometric method for the separation and determination of colchicine.** S. J. Smolenski, F. A. Crane and R. F. Voigt (Univ. of Illinois, Chicago, U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (5), 359–362.—Remove oils from the sample of colchicum seed (10 g in coarse powder) by Soxhlet extraction with light petroleum (100 ml). Extract the colchicine with a 1% soln. of ethanol in ethyl acetate (100 ml). Evaporate the extract to dryness and wash the residue on to a chromatographic column of alumina (20 g) with methanol (3 ml) followed by benzene (100 ml). Elute the lowest of the three coloured bands with $CHCl_3$ (100 ml) followed by 4% methanol in $CHCl_3$ (200 ml) and 10% methanol in $CHCl_3$ (200 ml). Evaporate the combined eluates to dryness, dissolve the residue in $CHCl_3$ (50 ml) and transfer to a column of silicic acid (15 g). Elute in turn with $CHCl_3$ (100 ml), 1% methanol in $CHCl_3$ (125 ml) and 2% methanol in $CHCl_3$ (50 ml) and reject the eluates. Elute the colchicine with 10% methanol in $CHCl_3$ (150 ml), evaporate and dry at 105° for 16 hr. To a portion of the residue containing < 0·4 mg of colchicine, add conc. $HNO_3$ (0·1 ml); after 30 min. add NaOH soln. (2·5 ml), dilute with $H_2O$ to 50 ml and measure the extinction at 350 mμ after a further 15 min. The results by this procedure are about 8% lower than those by a gravimetric method. The coeff. of variation, calculated from 12 replicate determinations, is ≃ ± 7%. A. R. ROGERS

1068. **Estimation of total alkaloids in tincture of ephedra.** S. K. Srivastava, R. C. Nandi and G. K. Ray (Central Drug Res. Inst., Lucknow, India). *Indian J. Pharm.*, 1958, **20** (5), 131–133.—Two methods are described that avoid the unbreakable emulsions formed during the extraction with $CHCl_3$ - ether (1:3) according to the method of the Indian Pharmacopoeia 1955. The recovery of ephedrine in 4 experiments ranged from 99·2 to 103·6%. *Procedure*—Evaporate the sample (50 ml) to about 5 ml and cool. Either add dil. $H_2SO_4$ (25 ml), filter, wash the ppt. with 0·1 *N* $H_2SO_4$, extract the combined filtrate and washings with light petroleum (boiling-range 40° to 60°) (2 × 20 ml) and reject the extract; or add 5% Pb acetate soln. (25 ml), filter, wash the ppt. with dil. $H_2SO_4$ (10 ml) and 0·1 *N* $H_2SO_4$ (5 × 10 ml), add excess of dil. $H_2SO_4$ to the filtrate, filter, and reject the ppt. Saturate the aq. soln. with NaCl, make alkaline with aq. $NH_3$ and extract with ether (135 ml in portions). Wash the combined ethereal extracts with satd. aq. NaCl soln. (5 ml) and filter. Evaporate the filtrate to dryness, dissolve the residue in 0·1 *N* $H_2SO_4$ (20 ml) and titrate with 0·1 *N* NaOH. A. R. ROGERS

1069. **Adaptation of the methyl orange method for the determination of reserpine.** E. A. De Felice (Biol. Sciences, New England Coll. of Pharmacy, Boston, Mass.). *Experientia*, 1958, **14** (4), 159–160.—The method described is applicable to pharmaceutical preparations and biological materials. Reserpine is extracted from slightly acid soln. with 1:2-dichloroethane, and methyl orange is added to form a complex in the organic solvent. The colour is developed by addition of acid ethanol, and the extinction, which is proportional to concn. of reserpine ranging from 0·3 to 15 μg per ml of 1:2-dichloroethane, is measured at 528 mμ after 5 min. By this rapid and simple procedure over 92% recovery is attained, and the method has been used to estimate the disappearance of reserpine from the plasma in dogs. P. NICHOLLS

1070. **Determination of reserpine in pharmaceutical preparations.** G. L. Szendey. *Arch. Pharm., Berlin*, 1958, **291**, 215–218.—From preparations containing both reserpine and theophylline, the two alkaloids are extracted with $CHCl_3$ and the extract is shaken with 1% NaOH soln. to remove theophylline. After the extract has been dried with anhyd. $Na_2SO_4$, reserpine is titrated with standard toluene-*p*-sulphonic acid in $CHCl_3$, with dimethyl yellow as indicator. The error is $\pm 2{\cdot}5\%$. Water and ethanol interfere with the titration.
M. H. Sawistowska

1071. **Determination of sparteine and paper chromatography of preparations of common broom, *Sarothamnus scoparius* (L.) WIMM.** H. Schindler (Betriebslabortorium der Firma Dr. Willmar-Schwabe, Karlsruhe-Durlach). *Dtsch. ApothZtg*, 1958, **98** (33), 832–834.—A new method for the determination of sparteine in tinctures consists in extracting the alkaloid with ether from the steam-distillate and pptg. it as sparteine dipicrate. The procedure for the chromatographic confirmation of sparteine is given. Flowers and branches of the investigated plants contain almost no sparteine but a flavone scoparin is present.
M. H. Sawistowska

1072. **Microchemical reactions for the detection of the glycoalkaloid tomatine.** E. A. Tukalo. *Sb. Nauch. Rabot Dnepropetr. Med. Inst.*, 1956, **1**, 347–349; *Ref. Zhur., Khim., Biol. Khim.*, 1958, (11), Abstr. No. 13,300.—An 80% soln. of $H_2SO_4$ and a freshly prepared soln. of $NH_4VO_3$ in $H_2SO_4$ (1:1000) are recommended as reagents for the detection of tomatine in vegetable matter. A few drops of reagent are added to previously dried sections of the material. It is shown that the content of tomatine is considerably higher in tomato flowers than in other organs.
K. R. C.

1073. **A direct method of determining salicin by paper chromatography.** A. Persson (Royal School of Forestry, Stockholm, Sweden). *J. Chromatography*. 1958, **1** (3), 269–270.—For the semi-quant. determination of salicin in aq. soln. (0·5 to 8%), develop on Whatman No. 1 paper with butanol - acetic acid - $H_2O$ (4:1:5) for 16 hr. by the ascending technique, dry, spray with 2 *N* $H_2SO_4$ and heat to 75° in a thermo-desiccator. After 1 or 2 min., salicin appears as pink spots at an $R_F$ value of 0·57.
A. R. Rogers

1074. **Anthraquinones. X. A simple paper-chromatographic method for identification of 'anthraquinone' drugs.** H. Auterhoff and N. Maiss (Pharm.-Chem. Inst., Tübingen Univ., Germany). *Dtsch. ApothZtg*, 1958, **98** (26), 659.—The procedure consists in impregnating a suitable paper with $Na_2CO_3$ soln. and using a mixture of ethanol and $H_2O$ as solvent in the descending method; 14 hr. are required for the solvent to travel 45 cm. The drugs for analysis are either directly extracted with methanol or first hydrolysed with a mixture of methanol and HCl, then diluted with $H_2O$ and extracted with ether. Aloes, cascara, frangula, Chinese rhubarb, rhapontic rhubarb, senna, *Morinda citrifolia*, and phenolphthalein tablets were analysed.
M. H. Sawistowska

1075. **The polarographic determination of santonin in mixtures.** Yung-ming Yu. *Acta Pharm. Sinica*, 1957, **5** (4), 355–360.—Dissolve about 15 mg of santonin in 25 ml of abs. ethanol. To 4 ml of this soln. add 4 ml of 0·2 *M* $CaCl_2.H_2O$, 2 ml of 0·05% gelatin soln. and 2 ml of water and polarograph.
Sci. Abstr. China

1076. **Colorimetric determination of santonin. I. Ferric chloride method.** Chu-ning Tsao and Shih-yen Sha. *Acta Pharm. Sinica*, 1957, **5** (3), 225–234.—The ferric chloride colour test for santonin has been adapted for its quant. determination.
Sci. Abstr. China

1077. **Asafoetida and its standards of purity.** T. V. Mathew (Central Food Laboratory, Calcutta). *J. Instn Chem., India*, 1958, **30** (3), 153–156.—The following standards for asafoetida are suggested by the Central Committee for Food Standards—ethanolic extract not to give a ppt. with neutral Pb acetate; ethanol-insol. solids $\geqslant 70\%$; matter sol. in light petroleum at 40° to 60° (volatile oils) $\leqslant 5\%$; ratio of total S to volatile oils $\leqslant 0{\cdot}25$; fibre content $\geqslant 10\%$; ash content $\geqslant 10\%$. Of 11 genuine samples, none satisfied all these requirements. The only requirement satisfied by all samples was that relating to fibre. Moisture content varied from 3 to 20%, and a standard should be established for this. More samples must be analysed before satisfactory standards can be suggested.
M. D. Anderson

1078. **A microchemical method for the determination of 'Sakaguchi-positive' antibiotics [guanidine derivatives]. II. Primycin.** I. Szilágyi and I. Szabó (Antibiotics Dept., Inst. Expt. Med. Res., Hungarian Acad. Sci., Debrecen). *Arzneimittel-Forsch.*, 1958, **8** (6), 333–336.—N-Bromosuccinimide was found to be suitable for the preparation *in situ* of hypobromite for use in the Sakaguchi test for guanidine derivatives. The modified procedure was employed to estimate streptomycin, viomycin and primycin, and was also found suitable for the chromatographic identification of these antibiotics and of arginine. (*Cf. Anal. Abstr.*, 1958, **5**, 3122.)
A. G. Cooper

1079. **New titrimetric determination of N-alkylamino derivatives of phenothiazine.** G. Dušinský and O. Lišková (State Inst. for Control of Drugs, Bratislava, Czechoslovakia). *Chem. Zvesti*, 1958, **12** (4), 213–220.—Conditions for the oxidimetric determination of chlorpromazine (**I**), diethazine (**II**) and promethazine (**III**) have been investigated. In an acid soln. the derivatives react with $Ce(SO_4)_2$ or with $KBrO_3$ losing one electron and forming a red semi-quinoidal free radical, which is decolorised when the second electron is lost. The course of the titration can be controlled visually, potentiometrically or by the dead-stop method (200 mV). *Procedure*—Dissolve the sample (0·01 to 0·1 g) in $H_2O$ (20 to 30 ml) and titrate in 0·1 *N* to *N* $H_2SO_4$ for (**I**), *N* to 1·5 *N* $H_2SO_4$ (for **II**) or 1·2 *N* to 1·4 *N* $H_2SO_4$ (for **III**) with 0·1 *N* $Ce(SO_4)_2$, or in 2 *N* to 3 *N* HCl with 0·1 *N* $KBrO_3$ after the addition of KBr soln. (10%) (5 ml). The method is suitable for the determination of **I**, **II** and **III** in pharmaceuticals. Caffeine, amphetamine, codeine, barbiturates, lactose and starch do not interfere.
J. Zýka

1080. **Quantitative determination of naphazoline and its hydrolytic degradation products.** M. J. Stern (Coll. of Pharm., Rutgers, State Univ., Newark, N.J.). *Drug Standards*, 1958, **26** (5), 158–160.—This method for the determination of naphazoline (**I**), N-1-(naphthylacetyl)ethylenediamine (**II**) and 1-naphthylacetic acid is based on the procedure

of Schwartz *et al.* (*cf. Anal. Abstr.*, 1957, **4**, 1964). Chloroform alone is used to elute **I** and **II** together from the Celite 545 - borate buffer column; **I** is determined in the eluate by absorptiometry (*cf.* Slack and Mader, *Anal. Abstr.*, 1958, **5**, 3509) and **I** plus **II** spectrophotometrically at 284 mμ.

A. R. Rogers

1081. **The micro-identification of medicaments, particularly by means of the polarising microscope.** A. Wickström (Inst. of Pharmacy, Oslo Univ.). *Ann. Pharm. Franç.*, 1958, **16** (2), 133–142.—The use of the polarising microscope for the determination of refractive indices as a means of identifying crystals of drugs is described. Examples are given from several sympathomimetic drugs and local anaesthetics. E. J. H. Birch

1082. **The detection of 1-ethynyl*cyclo*hexyl carbamate [ethinamate] in toxicological specimens and its separation from a mixture of other drugs.** G. R. Nakamura (406th Medical General Lab., APO 343, San Francisco, Calif.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (5), 366–368.—Ethinamate (**I**) is extracted by $CHCl_3$ from aq. soln. at pH 8. It gives an orange colour with conc. $H_2SO_4$; colour reactions with 9 other reagents are listed. Separation from several common pharmaceutical substances (including bromvaletone and morphine) is achieved by ascending chromatography either on paper with *iso*amyl alcohol - aq. $NH_3$ (*d*, 0·880) (3 : 1) as developing solvent or on paper impregnated with glacial acetic acid with *iso*amyl alcohol or *cyclo*hexane as solvent. **I** is detected on the chromatograms with an ammoniacal $AgNO_3$ spray. Values of $R_F$ are given. A. R. Rogers

1083. **Paper chromatography of iodine-131-labelled iodopropylidene glycerol with autoradiographic identification.** G. F. Hoffnagle, C. N. Sideri and A. Osol (Philadelphia Coll. of Pharm. and Sci., Pa.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (5), 315–317.—The paper-chromatographic characteristics of iodopropylidene glycerol isomers (**I**) in 12 different solvent systems have been investigated. The upper phase of an equilibrium mixture of *n*-butanol - 0·2 *M* phosphate buffer of pH 7·2 was preferred for the separation of **I** from other compounds of iodine. Location of $^{131}I$-labelled **I** was effected by Geiger - Müller counting and by autoradiography. A. R. Rogers

1084. **Determination of phenylcinchoninic acid (cinchophen) in the presence of acetylsalicylic acid, sodium bicarbonate, caffeine, hexamine and lactose.** W. Kamp and J. W. Kampman (Farm. Lab., Rijksuniv., Utrecht, Holland). *Pharm. Weekbl.*, 1958, **93** (11), 545–549.—Several methods investigated gave erratic results. A modification of Emery's method (*cf. J. Amer. Pharm. Ass., Sci. Ed.*, 1928, **17**, 18) *i.e.*, addition of glacial acetic acid to the dried residue after sodium carbonate extraction, provided reproducible results.

P. Rentenaar

1085. **Determination of small quantities of chlorhexidine in pharmaceutical preparations.** A. Holbrook (I.C.I. Ltd., Pharm. Div., Hexagon House, Blackley, Manchester 9). *J. Pharm. Pharmacol.*, 1958, **10** (6), 370–374.—*Procedure*—Remove the fatty excipients from creams and ointments by extraction with $CHCl_3$ from an acid soln. Extract chlorhexidine from lozenges by shaking an aq. NaOH soln. with ether, then extracting the soln. with aq. HCl. Simple and compound aq. soln. of chlorhexidine require no preliminary treatment. Dilute an aliquot of the aq. soln. (containing 1·5 mg) with $H_2O$ to 80 ml, add 20% cetrimide soln. (5 ml) and render alkaline with *N* NaOH. Mix well, add *iso*propyl alcohol (1 ml) and place in a water bath at 20° ± 2°. Add alkaline hypobromite soln. (containing 1% of available Br in *N* NaOH) (2 ml), dilute with $H_2O$ to 100 ml, mix and replace in the water bath. After 25 min., measure the extinction at 480 mμ in 1-cm cells against a reagent blank.

A. R. Rogers

1086. **Chromatographic separation of *m*-aminophenols from sodium 4-aminosalicylate.** M. Vietti-Michelina (Univ., Piazza Arbarello 8, Turin, Italy). *Z. anal. Chem.*, 1958, **162** (2), 116–117.—The method of separation depends on the fact that *m*-aminophenol is soluble in aq. *n*-butanol whilst Na 4-aminosalicylate is insoluble. *Procedure*—Apply one drop of a mixture of the two substances, in alcohol soln., 2 cm from the end of a strip of Whatman No. 1 paper 5 cm wide and 20 cm long. Elute the paper with water-satd. *n*-butanol. After 3 hr. the front will have risen 15 cm. The Na 4-aminosalicylate will remain almost at the spot where the test soln. was applied, whilst the *m*-aminophenol will have moved to within 2 cm of the front. The Na 4-aminosalicylate may be detected by a violet fluorescence given in u.v. light, or by an intense brown colour given after spraying the paper with $FeCl_3$ soln. The limit of detection is 10 μg.

D. F. Phillips

1087. **Coulometric micro-determination of mixtures of sodium 4-aminosalicylate and *m*-aminophenol (in substance and tablets).** K. Kalinowski and Z. Sykulska (Inst. Pharm. Chem., Lodz, Poland). *Acta Polon. Pharm.*, 1957, **14** (4), 255–260.—The substance (0·1 g) is dissolved in methanol (2 ml) and made up to 100 ml with peroxide-free ether. After 5 min., the ppt. (Na 4-aminosalicylate) is filtered off, washed with ether, dried, dissolved in water and made up to 1 litre. This soln. (10-ml aliquots) is used in the coulometric determination with electro-generated bromine. The ether filtrate, containing the *m*-aminophenol, is evaporated to dryness and the residue is dissolved in warm water and made up to 50 ml. Aliquots (10 ml) are determined coulometrically as for the aminosalicylate. The results are accurate to within ±0·5% for Na 4-aminosalicylate and ±0·2% for *m*-aminophenol, on 1 mg of substance. B. Lake

1088. **Application of anhydrous titrimetry in propionic acid media to the determination of [quaternary] ammonium halides.** C. Hennart and E. Merlin (Lab. de Vitry, Seine, France). *Chim. Anal.*, 1958, **40** (5), 167–169.—The sample, *e.g.*, cetyltriethylammonium bromide, is dissolved in a 0·1 *M* soln. of mercuric propionate, which is then titrated potentiometrically to a purple end-point (Metanil yellow indicator) with 0·1 *M* $HClO_4$ in anhyd. propionic acid. Residual moisture in the reagents is removed by treatment with a few decigrams of propionic anhydride. The end-point is sharp and occurs exactly in the centre of the inflection of the titration curve. The error is < ± 0·5%. W. J. Baker

1089. **Reaction between quaternary ammonium surface-active agents and methyl orange.** G. Nebbia (Bologna Univ., Italy). *Chim. e Ind.*, 1958, **40**,

638–640.—Methyl orange forms a salt with equimolar amounts of quaternary ammonium surface-active agents, which is sol. in fat solvents but of low solubility in $H_2O$. The surface-active agent may be determined by extraction of the reaction mixture with $CHCl_3$ and measurement of the extinction at 425 m$\mu$. L. A. O'Neill

1090. **Microbiological assay of mercurials in pharmaceutical products.** D. V. Carter and G. Sykes (Microbiological Div., Standards Dept., Boots Pure Drug Co. Ltd., Nottingham, England). *Analyst*, 1958, **83**, 536–539.—In the tube dilution method the medium is a peptone - glucose - phosphate medium with bromocresol purple indicator and the culture is a strain of *Escherichia coli* grown in nutrient broth at 37° and diluted with sterile saline. The control soln. has a formulation similar to that of the test material but without mercury, the theoretical amount of the mercurial under assay being added in the least vol. of solvent. Control and sample soln. are diluted suitably and further dilutions are then made in 10% stages to span the expected end-point. Portions (5 ml) of each dilution in 4 tubes containing 5 ml of the medium are inoculated with 0·2 ml of the diluted culture and are incubated at 37° for 48 to 72 hr. The end-point is the greatest dilution at which no growth occurs. In the rapid method a standard drop (0·03 ml) of the undiluted culture is added to each tube and incubation is for 4 hr. With initially turbid samples the end-point is shown by the indicator. In the plate-diffusion method, which follows in principle the Heatley cup assay for penicillin, the medium is peptone - Lab - Lemco - agar and the culture is a growth of *Staphylococcus aureus* in nutrient broth. As before, a control soln. of formulation similar to that of the test material is used. The procedure is that of the B.P. microbiological assay of penicillin. Satisfactory zone responses are obtained with concn. of 10 and 2 p.p.m. of phenylmercury compounds and thiomersal. This assay is sensitive down to $\simeq$ 1 p.p.m.
A. O. Jones

1091. **Alcohol determination in sweet orange-peel tincture.** E. Ehrenstein and L. F. Tice (Philadelphia Coll. of Pharm. and Sci., Philadelphia, Pa.). *Drug Standards*, 1958, **26** (5), 161–164.—To determine ethanol in liquids containing volatile substances, dilute the sample to contain $\simeq$ 25% of ethanol, saturate a 50-ml aliquot with NaCl, extract with light petroleum (3 × 25 ml), wash the combined extracts with satd. aq. NaCl soln. (3 × 10 ml), add the washings to the aq. phase, distil, and complete the determination by the method of the U.S.P. XV. A. R. Rogers

1092. **Determination of bismuth in pharmaceutical preparations and some prescription formulae with EDTA (disodium salt).** L. Krówezyński and A. Banaszek (Z Zakładu Chem. Farm. A. M. w Luklinie). *Acta Polon. Pharm.*, 1957, **14** (5), 337–343.—The sample is dissolved in an appropriate solvent (10 ml), *e.g.*, 5 *N* $HNO_3$ for inorganic preparations and 5 *N* HCl for organic samples; in some cases it is necessary to use a mixture of acetone, NaOH and tartaric acid. To the soln. are added 8% thiourea soln. (2·5 ml), enough borate buffer to adjust the pH to approx. 5·2 and a drop of methylene blue as indicator, and the soln. is titrated with 0·01 *M* EDTA (disodium salt). All samples were checked gravimetrically and the average variation in results was found to be 0·5%. The use of this method greatly decreases the time required for the determination. B. Lake

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*See also Abstracts*—**957**, Determination of peroxides in ether. **959**, Analysis of polyoxypropylene glycols. **1099**, Microscopic identification of stramonium seeds.

## Food

*Foods and food additives, beverages, edible oils and fats, vitamins.*

1093. **International Commission for Uniform Methods of Sugar Analysis. List of recommendations adopted at the 12th Session of ICUMSA at Washington, D.C., June 2-6, 1958.** *Int. Sugar J.*, 1958, **60**, 263–267.—The recommendations are reproduced alone without referee reports, discussions, etc.; the full official text of the proceedings will be published as a bound volume later.
Sugar Ind. Abstr.

1094. **Determination of invert sugar by the Lane - Eynon method using potassium ferrocyanide.** A. S. Sushkova. *Ukr. Khim. Zhur.*, 1958, **24**, 251–253.—A refinement of the Haddon modification of the Lane - Eynon method is described. The formation of $Cu_2O$, which obscures the end-point in the presence of methylene blue indicator, is prevented by addition of $K_4Fe(CN)_6$ in powder form. For calculation of the results by the standard Lane - Eynon tables, the titration figures must be multiplied by the correction factor 1·213 or the concn. of the Fehling's soln. altered accordingly. In the latter case, the procedure is—12·5-ml aliquots of both soln. I (84 g of $CuSO_4.5H_2O$ per litre) and soln. II (419·6 g of Rochelle salt and 121·3 g of NaOH per litre) are mixed in a 250-ml flask; 0·4 g of powdered $K_4Fe(CN)_6$ is added and the flask is rotated to dissolve the powder. About one-fifth of the sugar soln. required for the complete titration is measured into the flask from a burette, the flask is heated to boiling, and the mixture is titrated within 2 to 2·5 min. with the rest of the sugar soln. Methylene blue (2 to 3 drops of a 1% soln.) is added just before the end of the titration. For reproducible results, the concn. of the sugar soln. should be such as to require 15 to 30 ml per 25 ml of the modified Fehling's soln. Sugar Ind. Abstr.

1095. **Determination of reducing sugars in paper chromatograms by means of ceric sulphate.** H. Thaler. *Nahrung*, 1958, **2**, 111–116.—The usual analysis with copper reagents is liable to error owing to autoxidation of the cuprous oxide formed in finely divided or colloidal amounts. This is obviated by application of the ferricyanide and ceric sulphate method. The cut-out portions of the paper chromatogram (several strips) are extracted in a small glass funnel with 15 ml of cold water divided into 3 or 4 portions. The filtrate is put into a Hagedorn tube (100 mm × 34 mm), to which are added 2 ml of alkaline $K_3Fe(CN)_6$ soln. [5 g of $K_3Fe(CN)_6$ and 10·6 g of anhyd. $Na_2CO_3$ in 1 litre]. The tube is covered and heated in a boiling-water bath for 15 min., cooled in running water for 3 min. and, after adding 1 ml of 18 *N* $H_2SO_4$ and 7 drops of Setopalin C soln., the soln. is immediately titrated with 0·0025 *N* $Ce(SO_4)_2$ until the colour changes from yellow to a clear gold - brown. A blank test should also be made.

The amounts of sugar corresponding to 1 ml of the $Ce(SO_4)_2$ soln. are, in μg, maltose 101·6, lactose 120·2, glucose 82·7, mannose 80·7, galactose 103·0, xylose 82·0, fructose 77·6 and arabinose 89·1.

SUGAR IND. ABSTR.

1096. **Chemical analysis of honey: qualitative determination, by paper partition chromatography, of six saccharides.** M. Galanti and J. Wernimont (Inst. Agronom. de l'État à Gembloux, Belgium). *Rev. Ferment.*, 1958, **13** (2), 55–58.—A method for the clear separation of glucose, fructose, sucrose, maltose, melibiose and raffinose, with a descending solvent technique, is described. The developing solvent is benzene - *n*-butanol - pyridine - $H_2O$ - acetic acid (1:5:3:3:1). The colour developer is a mixture of 2% ethanolic soln. of diphenylamine and aniline and 85% $H_3PO_4$ in the ratio 5:5:1. The optimum concn. of honey in a soln. to be analysed by this method is 8 to 10%.

J. V. RUSSO

1097. **Analysis of Indian honey.** A. K. Mallik (Central Food Laboratory, Calcutta). *J. Instn Chem., India*, 1958, **30** (3), 171–173.—Samples of honey from different parts of India were examined with particular reference to standards established by the Food Adulteration Act, 1954. Known methods were used for the determination of moisture, ash, and reducing sugars and sucrose. Optical rotation, diastase, acidity and colour were also investigated. M. D. ANDERSON

1098. **Quantitative determination of copper and iron in starch syrup.** H. W. Ardelt and P. H. Opel (Inst. für Ernährungsforsch., Potsdam-Rehbrücke, Germany). *Z. LebensmittUntersuch.*, 1958, **107** (3), 232–236.—*Procedure*—Add, dropwise, a mixture of the sample (10 to 15 g) with 30% $H_2O_2$ (50 ml) to boiling $H_2SO_4$ (10 ml in a Kjeldahl flask). Evaporate the bulk of the acid, dilute the residue with 10 to 15 ml of water, boil for 2 to 3 min., and transfer to a 50-ml flask. Neutralise the soln. (to bromophenol blue), add 0·5 ml of 10 *N* NaOH, 0·2 *M* EDTA (tetrasodium salt) (12·5 ml), triethanolamine (2 ml), 2 *M* ethylenediamine (1·25 ml) and 3 *M* $Na_2SO_4$ (0·3 ml). After being made up to 50 ml, the soln. is ready for the polarographic determination. The $E_{\frac{1}{2}}$ values for Cu and Fe are −0·53 and −0·90 V, respectively. The procedure enables 0·5 μg of Fe or Cu per ml to be determined, and is of the same order of accuracy as the colorimetric method of Lindemann (*cf. Anal. Abstr.*, 1956, **3**, 3191). P. S. ARUP

1099. **Detection by polarisation microscopy of fragments of seeds of *Datura stramonium* in buckwheat flour.** A. T. Czaja (Botanische Inst. der Tech. Hochschule, Aachen, Germany). *Z. LebensmittUntersuch.*, 1958, **107** (3), 217–220.—The histological characteristics of the flour and the contaminating seeds are described and illustrated by photomicrographs. The presence of the seeds is principally revealed by the sharply-defined stellate structures of the testa and by the thick-walled, slightly rounded cells of the endosperm. The presence of the (poisonous) seeds has occasionally been detected in South African buckwheat.

P. S. ARUP

1100. **Determination of calcium in meat and bone meal.** M. Lapidus and E. F. Mellon (Eastern Reg. Res. Lab., U.S. Agric. Res. Service, Philadelphia, Pa., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 411–413.—The ether-extracted, ground and blended sample (1 g) is digested with $HNO_3$ followed by $HClO_4$ (risk of explosion through $HClO_4$ fumes reaching plastics or rubber tubing being avoided). After dilution to 50 ml a measured (1 to 5 ml) sample is passed into an Amberlite IRA-400 column, and is followed by water. The eluate (100 ml), treated with KCN, is brought to pH 12·5 with KOH, and treated also with Calcon indicator (200 mg) (*cf.* Hildebrand and Reilley, *Anal. Abstr.*, 1957, **4**, 2518; Barnard *et al.*, *Anal. Abstr.*, 1957, **4**, 2073) before titration with EDTA (disodium salt) soln. Recoveries of 99·1 to 100·5% of Ca (100 to 500 mg in 50 ml) in the presence of phosphate (molar ratio of Ca to P, 1·5) are reported.

A. A. ELDRIDGE

1101. **Phosphates and organic compounds of phosphorus in foods. II. Contribution to the analysis of polyphosphates.** J. Schormüller and G. Würdig (Inst. für Lebensmittelchemie und Lebensmitteltechnologie, Tech. Univ., Berlin, Germany). *Z. LebensmittUntersuch.*, 1958, **107** (5), 415–422.—A circular paper-chromatographic technique (*cf. Ibid.*, 1957, **105**, 397) is applied to the analysis of phosphate additives for sausage meat. Directions and data are given for the detection of monophosphate, polyphosphates (from pyro- to hepta-phosphate, and cyclic tri- and tetra-phosphates. A similar technique (*cf. loc. cit.*) is used for the analysis of a group of preparations containing 0 to 1% of P as monophosphate in combination with sugars and (in one case) organic compounds of N. The preparations containing 14 to 24% of P are based mainly on pyrophosphate, and contain small amounts of phosphates of greater complexity. The phosphate added as pyrophosphate to sausage meat is found, after cooking, to have been converted into monophosphate.

P. S. ARUP

1102. **Report on cryoscopy of milk.** W. F. Shipe (Dept. of Dairy Ind., Cornell Univ., Ithaca, N.Y., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 262–267.—A collaborative study showed that the Fiske and Hortvet cryoscopes give comparable results. The results are affected more by the accuracy of calibration than by the type of cryoscope used. A. A. ELDRIDGE

1103. **Brushite nodules and hydrogen swells in defective canned cheese paste.** D. F. Nelson (The Dominion Lab., P.O. Box No. 562, Dunedin, New Zealand). *Analyst*, 1958, **83**, 539–540.—Nodules of up to 5 mm diameter were found in defective cans of cheese paste. These contained 14·5% of P (present as $PO_4^{3-}$) and 19·1% of Ca. Less than 1% of the nodules were probably calcite; the remainder showed little variation and had a minimum refractive index of ≃ 1·539 and a maximum of ≃ 1·555, suggesting that the nodules consisted of brushite ($CaHPO_4.2H_2O$). X-ray powder diffraction examination confirmed their identity, and from this identification and the analysis it was deduced that the nodules consisted of 82% of brushite, together with cheese paste and water. It is probable that soluble phosphate added as an emulsifier had reacted with Ca in the cheese. The three cans contained 18, 33 and 40 ml of gas, respectively, and this was found to be 93·5% of H and 6·5% of $CO_2$. The pH of the cheese paste was 5·6, the interiors of the cans were etched, and one sample contained 1880 p.p.m. of Sn and 800 p.p.m. of Fe. A. O. JONES

1104. **Quantitative determination of diphenyl in citrus fruits.** B. Koether (Chem. Untersuchungsamt für das Saarland, Saarbrücken, Germany). *Z. LebensmittUntersuch.*, 1958, **108** (2), 158–163.—By this improved procedure, diphenyl is separated in a state of purity and determined spectrophotometrically in aq. soln. as a sulphonated product. *Procedure*—Mix the sample (200 g) intimately with water (200 ml) and steam-distil for 45 min. Make up the oily layer of the distillate to 5 ml with light petroleum, transfer 1 ml of this layer to the distillation flask, and shake with 100 ml of aq. 6% $KMnO_4$ soln. for 5 min. Steam-distil the mixture for 45 min. into a receiver containing 0·5 ml of light petroleum. Make up the upper layer to 5 ml with light petroleum, and transfer 1 ml of this layer to a test-tube provided with a glass stopper. Evaporate the solvent, and heat the residue with 1 ml of conc. $H_2SO_4$ to 105° for 10 min. Cool, make up to 100 ml with water, and determine the extinction value at 263 m$\mu$ in a 5-cm cell. As little as 0·15 mg of diphenyl per 100 g can be detected. Blank values amount to $<$ 0·1 mg per 100 g.

P. S. Arup

1105. **Methods for distinguishing sources of nutmeg or mace.** L. A. Lee and V. J. Caruso (E. Reg. Res. Lab., Agric. Res. Service, U.S. Dept. Agric., Philadelphia, Pa., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 446–449.—East Indian nutmeg and mace oils contain much greater quantities of phenols than West Indian oils. The phenols, distilled from the oils in steam, are coupled with a diazonium salt and the extinction of an aliquot of the dye is determined at 398 m$\mu$; West Indian oils afford values little higher than the blank. Another aliquot may be chromatographed on paper, when the origin of the coupled phenols is indicated by differences in colour and position of the zones with low $R_F$ values. In both cases eugenol predominates, but the conditions of the test decrease its obscuring effect.

A. A. Eldridge

1106. **Quantitative determination of traces of mercury, lead and arsenic in soup spices.** E. Krejcar (Res. Inst. of Fats and Oils, Prague). *Průmysl Potravin*, 1958, **9** (3), 162–163.—The method of Vašák and Šedivec for the determination of Hg with dithizone in the presence of EDTA (disodium salt) (*cf. Chem. Listy*, 1951, **45**, 10), their method for the determination of As with a soln. of Ag diethyldithiocarbamate in a pyridine medium (*Ibid.*, 1952, **46**, 341) and the dithizone method of Müller (*Z. anal. Chem.*, 1938, **113**, 161) for the determination of Pb in a medium containing tartrate, KCN and hydroxylamine have been found suitable for the determination of these elements in a $10^{-5}$ to $10^{-6}$ % concn. in soup spices.

J. Zýka

1107. **Analytical procedures for the determination of the colouring matter of ground paprika.** L. Benedek (Délalföldi Mezögazdasági Kisérleti Intézet, Szeged, Hungary). *Z. LebensmittUntersuch.*, 1958, **107** (3), 228–232.—The colour can conveniently be determined by measuring the extinction at 500 m$\mu$ of a benzene extract (obtained by shaking for 30 min. at room temp.) of the sample (previously dried at 65° to 70°). Owing to a cancellation of errors the results obtained by calculating the combined effect of the coloured constituents in terms of capsanthin are sufficiently accurate for routine purposes and in fair agreement with the results obtained by the separate chromatographic determination of the seven coloured constituents by the Cholnoky procedure.

P. S. Arup

1108. **Combination of wool-dyeing and chromatographic methods for detection of coal-tar dyes in foodstuffs.** S. N. Mitra and R. K. Chatterjee (Central Food Laboratory and West Bengal Public Health Laboratory, Calcutta). *J. Instn Chem., India*, 1958, **30** (3), 161–170.—Coal-tar dyes in foodstuffs are best extracted by the wool-dyeing method, the pH of the soln. being lowered gradually to get the optimum pH for each dye. The extract is concentrated, and submitted to chromatography on cellulose columns with 0·5 *N* HCl as eluent, and also on paper, with the circular method, with *iso*-butyl alcohol - ethanol - water (4:1:4) as solvent. The presence of a single dye is confirmed by paper chromatography with three spots, one of the sample, one of the pure dye, and one of a mixture of the two. Further corroboration is obtained by observing colour change with change of pH. When a mixture of dyes is present, a preliminary survey is made to determine the constituents and their approximate proportions; a control mixture of pure dyes of about the same composition is prepared, and chromatography by the three-spot method is carried out. The following water-sol. dyes were separated and identified by the method described—Brilliant black BN, Tartrazine, Sunset yellow FCF, Carmoisine, Ponceau 4R, Indigotine, Naphthol yellow S, Red 10B, Azo Geranine, Ponceau 2R, Metanil yellow, Orange I, Amaranth, Brilliant blue FCF, Wool green, and Erythrosine. Only the first six are permitted food dyes.

M. D. Anderson

1109. **Determination of carbohydrates in wort and beer. I.** K. Silbereisen, J. Bielig, F. Just and A. Kallmann (Inst. für Gärungsgewerbe, Berlin). *Brauerei, Wiss. Beil.*, 1958, **11** (5), 66.—The carbohydrates are adsorbed on a column of a mixture (1:1) of kieselguhr and activated carbon (Carboraffin) and then fractionally eluted by aq. ethanol of gradually increasing concn. The carbohydrates in the successive eluates can be satisfactorily separated by paper chromatography, and determined colorimetrically by means of a phenol - $H_2SO_4$ reagent. The carbohydrates found in lager beer include a (presumed) *iso*maltose, trisaccharides and other polymers of glucose, arabinose, xylose and ribose, but no hexoses. Boiled worts contain the above-mentioned disaccharide in measurable amounts, but no pentoses.

P. S. Arup

1110. **Potentiometric titration of the total acid content of wines and liqueurs.** T. Salo, R. J. Peltonen and B. Ståhlström (Res. Lab., State Alcohol Monopoly, Helsinki, Finland). *Z. LebensmittUntersuch.*, 1958, **107** (5), 401–406.—Potentiometric titration is more accurate and convenient than titration with indicators. The sample (25 ml) is diluted to 100 ml, and titrated with 0·1 *N* NaOH to pH 7·0. The results (which are accurate within 1 mg of acid, calculated as tartaric acid) are not affected by variations in the content of ethanol or sugar, or by the presence of normal traces of $CO_2$; sparkling wines must, however, be freed from $CO_2$ by refluxing for 30 sec. before dilution and titration. In the titration of liqueurs, an interval of 1 min. must be allowed before taking readings towards the end of the titration. The use of a glass electrode renders the process practically independent of the presence of neutral salts.

P. S. Arup

1111. **Determination of *iso*propyl alcohol and *sec.*-butyl alcohol in fermented liquors.** L. Genevois and M. Lafon (Lab. de Chim. Biol., Fac. des Sci., Bordeaux). *Chim. Anal.*, 1958, **40** (5), 156–158.—The contents of *iso*propyl alcohol and of *sec.*-butyl alcohol in wines, similar fermented liquids and commercial amyl alcohol can be determined to within $\simeq \pm 10\%$ by the procedure given. The sample (50 ml) is distilled, and the alcohols in 20 ml of the distillate are oxidised with cold $K_2Cr_2O_7$ - $H_2SO_4$ mixture. The soln. is made neutral by addition of solid $(NH_4)_2SO_4.FeSO_4$, and is distilled until $\simeq$ 75 ml has been collected. After oxidation (cold $KMnO_4$ - $H_2SO_4$ soln.) of trace amounts of acetaldehyde, two further distillations are made to obtain the pure ketones ($\simeq$ 10 to 15 ml), the total content of which is determined by oxidation with 0·01 *N* iodine soln. in 5 *N* NaOH. The amount of acetone is determined separately in 0·5 ml of distillate by the colorimetric reaction with salicylaldehyde in alcoholic KOH at 50°; the extinction is measured at 546 mμ (ethyl methyl ketone does not interfere and can be determined by difference). The two ketones can be identified, in a distillate representing a 50-fold concn. of the original sample, by pptn. of their 2:4-dinitrophenylhydrazones, followed by paper chromatography of a $CHCl_3$ soln. of these (*cf.* Lynn *et al.*, *Anal. Abstr.*, 1956, **3**, 1771) and identification of each separated dinitrophenylhydrazone by its u.v. spectrum. Bordeaux wines contain from 20 to 30 mg of *iso*propyl alcohol and 10 to 15 mg of *sec.*-butyl alcohol per litre.

W. J. Baker

1112. **Detection of formaldehyde in wine and fruit juice with the use of chromotropic acid.** F. Paul (Höheren Bundeslehr u. Versuchsanstalten f. Wein- u. Obstbau, Klosterneuburg, Austria). *Mitt. Wein.- u. Obstbau, Wien, A*, 1958, **8** (4), 192–196.—The violet - red coloration given with chromotropic acid is specific for formaldehyde. *Procedure*—Steam-distil the sample (25 or 50 ml), and collect twice its bulk of distillate. Add 4 or 8 drops of the distillate to a 0·2% soln. of chromotropic acid in 72% (w/w) $H_2SO_4$, and heat the mixture at 60° for 10 min. If no colour appears, concentrate the distillate to 10% of its bulk by evaporation on a water bath, and then repeat the test. With the use of 8 drops of the conc. distillate, 1 mg of formaldehyde per litre of the sample can be detected by comparison with a blank test.

P. S. Arup

1113. **Determination of aldehyde [acetaldehyde] in wine.** E. Kielhöfer and H. Aumann (Weinforschungsinst., Trier, Germany). *Z. Lebensmitt-Untersuch.*, 1958, **107** (5), 406–413.—The accuracy of the current method (the iodimetric determination of the acetaldehyde - bisulphite formed in a distillate from the sample at pH 8·5) is greatly impaired by various side-reactions, which can be eliminated by the application of the following modification. Before distillation, the sample (10 to 20 ml) is acidified (with $H_3PO_4$) to a pH between 1 and 2, and the distillation is carried out in a Lieb - Zacherl apparatus (*cf.* Paul, *Anal. Abstr.*, 1954, **1**, 3118) in a current of air, and with gentle heating to the boiling-point during 15 min. The iodimetric titration of the aldehyde - bisulphite in the receiver is carried out after the soln. has been saturated with $NaHCO_3$ (*viz*, at pH 7 to 8 instead of at pH 9·6). A reliable and comparatively low blank value is obtained from a second distillation of the same soln. into fresh bisulphite soln., and a titration is repeated under the same conditions.

P. S. Arup

1114. **Detection of traces of artificial dyes in wine.** H. Thaler and F. H. Mühlberger (Dtsch. Forschungsanstalt für Lebensmittelchem., Munich, Germany). *Z. LebensmittUntersuch.*, 1958, **108** (1), 17–20.—This adaptation of Arata's method allows of the detection of 0·1 mg of dye (*e.g.*, Bordeaux red or Tartrazine) per litre of wine, in a single wool-dyeing operation. The wool used must be unbleached wool which has been defatted and steeped in 5% aq. $NH_3$ at 80° for 1 hr. *Procedure*—Dye the wool (4 successive 0·1-g pieces) in a boiling mixture of the sample (100 ml) and aq. 10% $KHSO_4$ soln. (10 ml). Rinse the wool and place it in boiling dil. HCl (100 ml of water plus 1·5 ml of conc. HCl). Boil for 10 min. and repeat the procedure until the (fresh) acid stays colourless. Rinse the wool and place it in a boiling mixture of aq. 1% acetic acid (50 ml) and aq. saturated Pb acetate soln. (10 ml). Boil for 10 min. to render the tannins and associated substances insol. in aq. $NH_3$. Rinse the wool repeatedly in warm water, and extract the dye with 1% aq. $NH_3$ (20 ml). Evaporate the extract down to 0·2 to 0·5 ml, and identify the dye by ascending paper chromatography and comparison with parallel runs of the suspected dyes.

P. S. Arup

1115. **Correlation of physical constants and chemical structure. Graphical statistical methods for the identification of mineral oils, glass, silicones, fatty oils and catalysts. IV. Analyses in the field of fatty oils.** H. I. Waterman, C. Boelhouwer and J. Cornelissen (Technol. Univ., Delft, The Netherlands). *Anal. Chim. Acta*, 1958, **18** (5), 481–492 (in English).—Methods available for the analysis of fatty oil mixtures include estimation of olefinic unsaturation by the hydrogen value and ozone addition, spectrophotometry, chromatography and molecular distillation. The cascading fractionating molecular still gives efficient separation of higher molecular fatty acid derivatives. Fatty oils can be converted into the corresponding hydrocarbons and analysed as such (*cf. Anal. Abstr.*, 1959, **6**, 980). High-pressure catalytic hydrogenation is quantitative but relatively drastic and yields hydrocarbons with one C atom less than the number in the hydrocarbon chain of the ester. A Grignard reaction followed by dehydration of the resulting tertiary alcohols and saturation of the olefinic bonds lengthens the hydrocarbon chains and gives yields of 70 to 90%. These procedures are useful for investigation of polymerisation of drying oils. Examples presented are the thermal polymerisation of linseed oil and tung oil fatty esters, the analysis of a copolymerisation product of styrene with methyl linoleate, and confirmation of the linear structure of the novolaks. For non-branched, non-conjugated and non-polymerised fatty oils a diagram is presented, illustrating a plot of specific refraction against iodine value, which enables mol. wt. and number of C atoms and number of double bonds per average molecule to be estimated. Iodine value, m.p. and refractive index are used to follow the course of fat hydrogenation. A graph of saturated acids (%) against iodine value for the selective hydrogenation of linoleic esters under a variety of conditions permits selection of the conditions to achieve a desired result. Graphical representation of conjugation and polymerisation of drying oils is possible by means of the iodine

value and specific refraction. The course of catalysed *cis - trans* conversion can be followed by the critical demixing temp. of the oil with a solvent. The styrene (%) in a copolymer with linseed oil can be determined from a refractive index *vs.* density diagram. G. Burger

1116. **Possibilities of using spectrophotometers (visible and ultra-violet) in the oils and fats and associated industries.** J. P. Wolff. *Chim. Anal.*, 1958, **40** (5), 159–166.—Applications of spectrophotometers for improving speed and accuracy of routine analyses are reviewed. Instruments operating between 400 and 700 mμ can be used to determine colour indices or coloured pigments of oils, p.p.m. of trace elements (*e.g.*, Cu and Fe) simultaneously, and contents of P, antioxidants, vitamins, cholesterol or cholorophyllins. Those operating between 230 and 350 mμ can be used to determine the state of oxidation of crude oils or fats, to follow oxidation stages during their refining or storage, to distinguish natural oils from refined products, to determine the content of unsaturated fatty acids in natural vegetable oils, and to identify and determine, for example, antioxidants (octyl gallate, butylated hydroxyanisole) in fatty acids, aryl hydrocarbons in essential oils, and alkylarylsulphonates in detergents. Some typical absorption bands and max. extinction wavelengths for some of the applications mentioned above are given. (28 references.)
W. J. Baker

1117. **Identification of oils and the detection of oil adulteration by differential infra-red spectroscopy.** J. G. Bartlet and J. H. Mahon (Food and Drug Directorate, Dept. Nat. Health and Welfare, Ottawa, Canada). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 450–459.—The oil sample, dissolved in $CCl_4$, is placed in a 0·5-mm NaCl cell, the reference oil soln. being placed in a matched cell, and both are placed in a double-beam i.r. spectrophotometer, whereby differences in extinction are measured. Differential spectra of rape-seed and other vegetable oils with olive oil are illustrated. About 10 to 20% of most fats and oils can thus be detected in olive oil; tea-seed oil is an exception. The purity of butter fat and the effect of dietary changes on the composition of rats' kidney fat may also be investigated by differential i.r. spectroscopy.
A. A. Eldridge

1118. **The determination of oil content and crude fat by the use of anhydrous ether, Skellysolve B and Skellysolve F.** D. A. Shearer and R. B. Carson (Chem. Div., Canada Dept. Agric., Ottawa). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 414–416.—Anhydrous diethyl ether extracts significantly more than does Skellysolve B from forages and silage, but less from wheat by-products; with oil-seeds and cereals there is little difference. In most cases less crude fat was extracted with Skellysolve F than with Skellysolve B or anhydrous diethyl ether. The values obtained with the petroleum solvents are preferred, since diethyl ether extracts more pigments from dried forages and more lactic acid from silages. A. A. Eldridge

1119. **Determination of ascorbic acid with thionine.** R. Charonnat and L. Bézanger-Beauquesne (Pharm. Centr. des Hôpit. de Paris). *Ann. Pharm. Franç.*, 1958, **16** (2), 96–102.—Ascorbic acid is determined by its reduction of thionine in acid soln. when irradiated. *Procedure*—Solids are triturated with $(NH_4)_2SO_4$ and extracted with saturated $(NH_4)_2SO_4$ soln. so that protein is not dissolved, and liquids are treated with $(NH_4)_2SO_4$ and filtered. In one tube are placed an aliquot of the extract containing between 25 and 250 μg of ascorbic acid (5 ml), water (5 ml) and thionine soln. (0·05 g per litre) (3 ml) and in other tubes standards of ascorbic acid in the range 25 to 250 μg, stabilised with KSCN, are treated similarly. To each tube is added 1 ml of 10 *N* $H_2SO_4$ and the tubes are then irradiated with sunlight or at 25 cm from a 75-W lamp. The unknown tube is then interpolated visually among the standards. A coloured extract may be compensated for by adding an equal vol. of extract to the standard and water to the unknown, immediately after irradiation and before reading. Dehydroascorbic acid can be determined by reduction with $H_2S$ and removal of $S^{2-}$ with $Hg(CN)_2$. A review with references of results obtained in various fields is given. E. J. H. Birch

1120. **Determination of vitamin C in fruit juice by paper chromatography. Application to black-currant juice.** J. Bergeret (Lab. de Chim. Appl. et d'Oenologie, Fac. des Sci., Dijon, France). *Ind. Aliment. Agric.*, 1958, **75** (4), 299–304.—The method of Strohecker *et al.* (*cf. Anal. Abstr.*, 1955, **2**, 2859) has been modified so that it is now quantitative. The sample (40 g) is ground with sand and 2 portions of 2% oxalic acid saturated with $CO_2$, and made up to 200 ml with ethanol. After filtration, two aliquots of the soln. are taken, one being reduced with $H_2S$ at a pH of 4 to 5. Ascending chromatography is used, the solvent being *n*-butanol - water (2:3) containing 2% of oxalic acid and traces of KCN. The pH of the soln. must be adjusted to 2·6 to 2·7 before chromatography. After chromatography, the spots are detected by spraying with ammonium molybdate soln. The soln. reduced with $H_2S$ contains the total ascorbic acid. The total vitamin C and the ascorbic acid are determined by Tillman's reagent, followed by back-titration with ferrous ammonium sulphate.
S.C.I. Abstr.

1121. **Polarographic method of estimating ascorbic acid in multivitamin preparations.** S. M. Deshpande and R. Natarajan (Dept. of Chem., Coll. of Sci., Benares Hindu Univ., India). *Drug Standards*, 1958, **26** (5), 181–184.—Mix the sample, containing $\lesssim$ 50 mg of ascorbic acid, with $KH_2PO_4$ buffer (pH 8·0) containing 1·5% of K citrate and 1·491% of KCl, dilute to 100 ml, filter, and record the polarogram of the filtrate from 0·1 to −0·15 V *vs.* the S.C.E. The diffusion current, corrected for the residual current and blank, is directly proportional to the concn. of ascorbic acid. Results are accurate to $\simeq \pm 3\%$. A. R. Rogers

1122. **Identification of stored-products insects by the micromorphology of the exoskeleton. VI. Adult and larval beetle mandibles.** M. M. Jackson, A. F. Ratay and E. J. Woznicki (Food and Drug Admin., Philadelphia, Pa., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 460–471.—Diagrams and photomicrographs of the mandibles of various grain and other beetles which commonly infest stored food are reproduced, and diagnostic characteristics are described.

**VII. Labral characteristics of adult beetles.** A. F. Ratay, M. M. Jackson and E. J. Woznicki. *Ibid.*, 1958, **41** (2), 472–478.—Diagrams and photomicrographs of the upper lip (labrum) of various adult beetles are reproduced, and diagnostic characteristics are described. A. A. Eldridge

1123. **The effect of natural and laboratory digestion on the microscopic appearance of certain animal hairs.** F. R. Smith (Div. Microbiol., Food and Drug Admin., Dept. of Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 479–480.—The damage suffered by rodent hairs, separated from pellets, by passage through the digestive tract is discussed and illustrated. Similar damage is suffered by hairs involved in the submission of cereal products to digestion by pancreatin or pepsin; boiling with HCl, however, did not have this effect.

A. A. Eldridge

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*See also Abstracts*—**960**, Determination of carbohydrates. **989**, Determination of dyes in foods. **1029**, Identification of hair. **1139**, **1140**, Detection of insecticide residues in fruit. **1159**, Determination of Wiley m.p. **1161**, Determination of $H_2O$ in grain.

## Sanitation

*Analysis of air, water, sewage, industrial wastes, industrial poisons.*

1124. **Neutron activation analysis of drinking water.** R. L. Blanchard, G. W. Leddicotte and D. W. Moeller (Oak Ridge Nat. Lab., Tenn.). *U.S. Atomic Energy Comm.*, Rep. A/CONF.15/P/796, 1958, 17 pp.—Drinking-water samples collected from various geographical locations throughout the U.S.A. were analysed by neutron activation. Each sample was analysed qual. and quant., and trace concn. of Al, A, As, Ba, Br, I, Mn, Rb and Th, none of which had been detected, or determined, by conventional water-analysis methods, were determined. In addition, Ca, Cu, Fe, Mg, K, Na, Sr, Zn, Cl and Si, usually determined by routine water-assay methods, were also determined. The radionuclides produced in the reactions of the stable isotopes with either thermal or fast neutrons in the Oak Ridge Nat. Lab. Graphite Reactor were used. Each analysis consisted of an irradiation of a portion from each sample in the reactor, and analysis either by direct radioactivity measurement or by radiochemical separation before the radioactivity measurement. Gamma counting, especially scintillation spectrometry, was used most frequently in making the radioactivity measurements. Gamma spectral analysis and half-life determinations were used to identify the induced radionuclides. In the quant. analyses, a comparator sample containing a known amount of the element of interest was irradiated and processed simultaneously with the unknown samples. Methods and procedures of sample irradiation and measurement of induced radioactivities are described.

Nucl. Sci. Abstr.

1125. **Determination of zinc in water.** V. P. Pavelkina and B. M. Donskoĭ (Kazan Aviation Inst.). *Zavod. Lab.*, 1958, **24** (5), 548.—To determine Zn ($< 0{\cdot}2$ mg per litre) in water containing Ca, Mg, Al, Fe and Cu by titration with 0·004 *N* EDTA (disodium salt), the interference of Ca and Mg is prevented by the addition of a 20% soln. of KF and that of Cu by addition of Na diethyldithiocarbamate, which does not react with Zn at pH 9 to 10, and the titration is carried out in the presence of an ammoniacal buffer soln. (containing 350 ml of 25% aq. $NH_3$ and 54 g of $NH_4Cl$ in 1 litre) to prevent the interaction of Fe and Al with EDTA, and with Chromogen black as indicator.

G. S. Smith

1126. **Determination of phosphorus in natural waters by extraction of molybdophosphoric heteropoly acids with butanol.** V. G. Datsko and A. D. Semenov (Hydrochem. Inst., Acad. Sci., USSR). *Izv. Akad. Nauk, SSSR, Otd. Khim. Nauk*, 1958, (3), 357–358.—A known method is applied. After addition of the molybdate reagent the coloured complex is extracted with butanol and reduced with $SnCl_2$ soln. The extinction is measured with a red filter.

G. S. Smith

1127. **Field laboratory methods for the determination of uranium in non-saline waters.** G. W. C. Milner and G. A. Barnett (A.E.R.E., Harwell, England). A.E.R.E. Report C/R2723, 1958, 16 pp.—The first method is applicable to waters with a content of U of 1 $\mu$g or more per litre, and can be completed in 20 min. The sample (1 ml) is evaporated in a platinum tray in the presence of Mg acetate, which acts as a carrier. After heating the residue to destroy organic matter, a mixture of NaF and $Na_2CO_3$ is added and a phosphor is prepared by a carefully controlled fusion. The phosphor is then examined in a fluorimeter (A.E.R.E. Type 1080A) and the result is compared with that from another 1-ml sample to which a standard addition of U has been made. In the second method, the sample (100 ml) is evaporated with Mg acetate and then ignited. The residue is dissolved in $HNO_3$ and the pH of the soln. is adjusted to between 6 and 7; EDTA (disodium salt) and 8-hydroxyquinoline are added and the U is extracted into $CHCl_3$. The whole extract is evaporated and after the addition of Mg acetate the procedure is similar to that in the first method. Waters containing as little as 0·1 $\mu$g of U per litre have been analysed.

G. J. Hunter

1128. **Methods for the determination of radionuclides in water in low concentrations.** B. Kahn and S. A. Reynolds. (Oak Ridge Nat. Lab., Tenn., U.S.A.). Nuclear Engng and Science Conf., Preprint, American Inst. of Chem. Engrs, New York, 1958, 19 pp.—Methods are described for concentration of radionuclides from large volumes of water and for subsequent separation and determination of radioactivity. Techniques employed include pptn., solvent extraction, distillation and ion exchange. A discussion is given of methods for the radioelements Zr, Nb, I, Ru and Sr. Scintillation spectrometers have been used for identification and quant. measurement of gamma-active radionuclides. Experimental arrangements are described, and the advantages of the method, such as speed and simplicity, are discussed. Factors limiting sensitivity are listed.

Nucl. Sci. Abstr.

1129. **Use of the gamma spectrometer for the identification of radionuclides in water.** G. R. Hagee, A. S. Goldin and C. P. Straub (Robert A. Taft Sanitary Engng Center, Cincinnati, U.S.A.). Nuclear Engng and Science Conf., Preprint, American Inst. of Chem. Engrs, New York, 1958, 15 pp.—The availability of commercial gamma spectrometers has greatly simplified the problem of identification of radionuclides. Experience in utilising this type of equipment for such identification in rain, cistern and surface waters is described. Data are presented showing differences obtained as

a function of water fraction, age of samples, etc. Some information on the applicability of this technique to quant. determinations of specific radionuclides is also included.

Nucl. Sci. Abstr.

1130. **Review of the literature of 1957 on sewage, waste treatment and water pollution.** Committee on Research, Federation of Sewage and Industrial Wastes Assoc. (Washington, D.C., U.S.A.). *Sewage Ind. Wastes*, 1958, **30** (5), 601–608.— Ninety references to analytical publications are given.

1131. **Photometric determination of very small traces of cyanide and thiocyanate in effluents from petroleum refineries.** F. Wagner (Lab. Scholven-Chemie A.-G., Gelsenkirchen, Germany). *Z. anal. Chem.*, 1958, **162** (2), 106–115.—A benzidine - pyridine procedure is described in which the effect of a large excess of sulphide was investigated. For satisfactory determinations it is necessary to know the exact content of $S^{2-}$ in order that, after the oxidation of sulphide by means of $KBr_3$, a constant excess of Br is present. In order to control this satisfactorily, a KBr soln. is saturated with Br and the effective Br content is determined. Colour development and absorption were investigated and max. absorption at 520 mμ was established. The limit of detection is $\simeq$ 0·005 mg of $CN^-$ per litre. For a content of $>$ 1 mg per litre of $CN^-$ plus $SCN^-$, a max. error of $\pm$ 5% is quoted; between 0·1 and 1 mg per litre of $CN^-$ plus $SCN^-$, the error is $\pm$ 10%; and for $<$ 0·1 mg per litre the error is $\pm$ 20 to 50%. The method is applicable to effluents with higher ammonia and alkaline sulphide contents as well as for strong caustic wash liquors and for $CN^-$-containing gases.

D. F. Phillips

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*See also Abstracts*—**972**, Determination of aromatic amines in air. **1148**, Portable mercury-vapour detector.

## Agricultural analysis

*Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.*

1132. **System for the determination of certain trace metals in crops.** W. D. Duffield (26, Park Crescent, Portland Place, London). *Analyst*, 1958, **83**, 503–508.—The ash of the sample (10 g) is triturated with water (0·5 ml), a 0·02-ml portion is reserved for determination of B, and the remainder is evaporated either with HF or repeatedly with HCl to separate Si. The extract of the residue with dil. HCl (0·5 or 1 ml) is the sample soln. Copper, Mo and Ni are determined by ascending paper chromatography with a solvent mixture of ethyl methyl ketone, HCl and water, and with ethanolic dithio-oxamide as spray reagent. Molybdenum is determined similarly with *n*-butanol and HCl as solvent mixture and a mixture of dithiol and thioglycollic acid in NaOH soln. as spray reagent, the colour band being then concentrated by upward diffusion of *n*-butyl acetate. If the Fe:Mo ratio is $>$ 100, Mo is determined colorimetrically with KSCN and $SnCl_2$, extraction of the mixture with *n*-butyl acetate and comparison of the extract with standards. For the determination of B, the chromatographic paper with spots of the sample soln. is sprayed with an ethanolic curcumin - oxalic acid soln., dried and, after upward diffusion of ethanol, subsequent upward diffusion of 0·5% NaOH soln. carries the excess of curcumin past the stationary band due to B. Comparison with simultaneously prepared standards is made immediately. For Mn the sample soln. (0·1 ml) is fumed with $H_2SO_4$, $HNO_3$ and $H_3PO_4$, the residue is oxidised to $KMnO_4$ by warming with $KIO_4$ and $AgNO_3$ and the colour is compared with that of standard soln. of $KMnO_4$. Zinc is determined colorimetrically by extraction of its dithizone complex with chloroform in the presence of $Na_2S_2O_3$.

A. O. Jones

1133. **Acid extraction and analysis of strontium-90 in soil. Recommended method for soil sampling.** G. H. Hamada, E. P. Hardy, jun., and L. T. Alexander (New York Operations Office, U.S.A.E.C.). *U.S. Atomic Energy Comm.*, Rep. HASL-33, 1958, 32 pp.—A procedure is described for the acid extraction and analysis of $^{90}Sr$ in soil. The methods of collection, sub-sampling, extraction, separation and counting are discussed, and the reproducibility and reliability of the analytical method are evaluated.

Nucl. Sci. Abstr.

1134. **Spectrophotometric determination of molybdenum, tungsten and vanadium in soils and plants. I. In soils.** J. H. Watkinson (Rukuhia Soil Res. Sta., Hamilton, N.Z.). *N.Z. J. Sci.* 1958, **1** (2), 201–219.—Detailed procedures are described for the determination of these elements in one aliquot of soln. The ashed sample is treated with HF and $H_2SO_4$ to remove Si, then Mo and W, complexed with citric acid, are separated from V and most elements that would interfere in their subsequent determination by passing the soil soln. through a cation-exchange resin. The Mo and W in the eluate are separated chromatographically on a column of anion-exchange resin and determined by the dithiol method. The V is eluted from the cation-exchange resin with 0·15% (0·5 vol.) $H_2O_2$ and determined by the tungstophosphate method. Under the conditions tested, for pure solutions and when known amounts have been added to soil samples, there is a satisfactory recovery of all three metals.

J. M. Jacobs

1135. **A detailed summative analysis of the crude fibre and nitrogen-free extractive fractions of roughages. II. The analysis of straw, hay, grass and mangold.** B. D. E. Gaillard (Lab. of Animal Physiol., Agric. Univ., Wageningen, Netherlands). *J. Sci. Food Agric.*, 1958, **9** (6), 346–353.—The procedure previously described (*J. Sci. Food Agric.*, 1958, **9**, 170), which includes the analysis of the polysaccharide fractions soluble in 0·5% ammonium oxalate soln., 5% KOH soln. and 24% KOH soln., has been applied to roughages (*a*) without delignification of the material, (*b*) with delignification after oxalate extraction, and (*c*) with delignification after extraction of the major part of the polysaccharides soluble in 5% KOH soln. Results are compared with those given by Harwood's method (*cf. Anal. Abstr.*, 1954, **1**, 3137). With materials of high lignin content ($>$ 11%, *e.g.*, straw), method (*a*) gives incomplete extraction of polysaccharides, whilst (*b*) and (*c*) are satisfactory, (*b*) being more rapid. With materials of moderate lignin content (6 to 11%, *e.g.*, hay, fresh and artificially dried grass, clover, etc.), (*b*) appears to cause slight losses in carbohydrate, but (*c*) is satisfactory. With material of low lignin content ($<$ 6%, *e.g.*, mangolds, turnips), delignification causes loss of carbohydrate, but (*a*) is satisfactory. Results are quoted and discussed.

R. E. Essery

1136. **Report on spectrophotometric determination of iodine in feeds.** D. Menschenfreund (Food and Drug Admin., Dept. of Health, Education and Welfare, Los Angeles, Calif., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (2), 225–230.—The sample (5 g) is heated with 10% ethanolic KOH soln. (10 ml), charred, and finally ashed at 525°. The filtered leachings are evaporated, neutralised with $H_2SO_4$, brought to a pH of 8 to 10 with NaOH, boiled, and treated with $KMnO_4$. After 30 min. at 100°, ethanol (2 ml) is added, the liquid is digested and then filtered, and the filtrate, kept at pH 8 to 10, is evaporated to 5 to 7 ml. It is then acidified (HCl) and shaken with *o*-xylene (15 ml) and 10% aq. KI soln. (0·5 ml), a second extraction being performed with 10 ml of *o*-xylene. The extinction of the xylene soln. is determined at 494 m$\mu$, and the amount of iodine (six times that originally present) is read from a standard curve. Recoveries of 90 to 100% of iodine (0·05 to 1 mg) are reported.

A. A. Eldridge

1137. **Spectrophotometric determination of total gossypol in cottonseed meal and cottonseed meats.** F. H. Smith (N. Carolina Coll. of Agric. and Engng, Raleigh, U.S.A.). *J. Amer. Oil Chem. Soc.*, 1958, **35** (6), 261–265.—A method is described for determining total gossypol in cottonseed and cottonseed meal by conversion into the dianilinogossypol. Ground meal (0·5 g) is treated with 2 ml of ethanol (830 ml of 95% ethanol and 0·2 ml of glacial acetic acid diluted to 1 litre with water) and allowed to stand at room temp. for 10 min.; 2 ml of aniline is then added and the mixture is heated on a water bath (not boiling) for 45 min. To the soln. is added 60 ml of $CHCl_3$. After shaking the soln. for 15 min. and then filtering under reduced pressure through Hyflo Super-Cel, the filtrate is diluted to 100 ml with $CHCl_3$, and 2 ml of this soln. is further diluted to 25 ml with $CHCl_3$. The intensity of the resulting soln. is determined spectrophotometrically at 440 m$\mu$. The total gossypol content is then determined by comparison with a standard curve. Results obtained with the method are slightly higher than those obtained with the *p*-anisidine or A.O.C.S. Tentative Method BA 8-55.

G. R. Whalley

1138. **3-Amino-1-propanol as a complexing agent in the determination of total gossypol.** W. A. Pons, jun., R. A. Pittman and C. L. Hoffpauir [S. Regional Research Lab. (U.S. Dept. of Agric.), New Orleans, La., U.S.A.]. *J. Amer. Oil Chem. Soc.*, 1958, **35** (2), 93–97.—The total gossypol in any cottonseed product is extracted by heating the sample for 30 min. at 100° with a soln. of 3-aminopropan-1-ol and excess of acetic acid in dimethylformamide. The extract is heated for 30 min. at 100° with aniline, cooled and set aside for 1 hr. The extinction at 440 m$\mu$ is measured against a similarly treated sample without the aniline as reference. The extinction of a reagent blank is subtracted, and the amount of gossypol determined by reference to a calibration curve. The extracts and reagents are stable for 1 week. The time required for the determination is 2 hr. The standard deviation (from 15 analyses) is $\pm$ 0·0084%. The results agree with those found by other accepted methods.

G. Burger

1139. **Routine quantitative detection of insecticide residues in fruit and vegetables.** W. Weinmann (Bundesanst. für Qualitätsforsch. pflanzl. Erzeugnisse, Geisenheim i. Rheingau, Germany). *Z. LebensmittUntersuch.*, 1958, **107** (6), 504–512.—Details are given of the extraction of the insecticide in preparation for the *Drosophila* toxicity test. *Procedure*—Homogenise a 1-kg sample with an equal amount of water, and separate 400 g of the mixture into press-juice and residue by pressure through cloth; extract the two portions separately with light petroleum (2 × 130 ml for the residue, and 1 × 100 ml, plus 5 ml of *n*-butanol, for the juice) with the use of a Starmix mixer. (For the extraction of Systox or metaSystox from the aq. portion it is necessary to use $CHCl_3$.) Allow the mixed light petroleum extracts (dried with $Na_2SO_4$) to evaporate spontaneously to dryness, mix the residue with sand, and extract with successive small amounts of methyl cyanide (total 10 ml) by grinding with a small pestle. Filter the extract into a separating funnel containing 20 ml of water, extract the aq. mixture with light petroleum (2× 25 ml), and allow these combined extracts to evaporate on the inside of the base and cover of a Petri dish, in preparation for the *Drosophila* test. Directions are given for the rearing of the flies. The kills (with 50 flies per dish) are observed at logarithmic intervals during 6 hr. The tests are conducted in parallel with a blank test on material known to be free from insecticide. The yields of purified insecticide residue vary from 0·05 to 1·0 mg per kg.

P. S. Arup

1140. **Determination of Mesulfane [N-methanesulphonyltrichloromethanesulphen-*p*-chloroanilide] residues in fruit.** H. J. Hardon, H. Brunink and E. W. van der Pol (Keuringsdienst van Waren, Amsterdam, Holland). *Z. LebensmittUntersuch.*, 1958, **107** (3), 215–217.—The procedure described depends on the (nearly quant.) production of *p*-chloroaniline (**I**) from Mesulfane (extracted from the fruit by $CCl_4$) on boiling for 1 hr. with 50% $H_2SO_4$. After separation by steam-distillation, the **I** is diazotised and coupled with N-1-naphthylethylenediamine. The extinction value of the coloration thus obtained, measured at 560 m$\mu$, is proportional to the Mesulfane content of the fruit. The Mesulfane content of various fruits, harvested 14 days after spraying, was 1 to 15 mg per kg. Blank values given by unsprayed fruit were $\simeq$ 0·025 mg. A similar coloration is given by *iso*-propyl phenylcarbamate and its chloro derivatives, which, however, can be distinguished from Mesulfane by a colour reaction with hypochlorite (not given by **I**) after the treatment with $H_2SO_4$.

P. S. Arup

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*See also Abstracts*—**975**, Analysis of triphenyltin compounds. **1118**, Extraction of oil from forage and silage.

## 5.—GENERAL TECHNIQUE AND APPARATUS

### General

1141. **Completely automatic titration unit for process use.** J. F. Brown and R. J. Weir (I.C.I. Ltd., Billingham Div., Res. Dept., Co. Durham, England). *Analyst*, 1958, **83**, 491–503.—This unit for continuous operation on chemical plant takes the sample, delivers and dilutes a measured vol., fills the burette, titrates the liquid to a pre-set pH end-point, records the burette reading and repeats the sequence after a set time-interval. Control of

liquid flow is by solenoid-operated glass valves, vol. of liquid are measured by level electrodes, and the level of liquid in the burette is measured by means of a photocell and lamp unit that travels up the burette and detects the meniscus. The completion of any operation moves a controlling Uniselector to its next position. In titres of 10 ml with $N$ acid and alkali the standard deviation is $\pm$ 0·04 ml.
A. O. Jones

1142. **Coulometric titrations in molten lithium chloride - potassium chloride eutectic.** H. A. Laitinen and B. B. Bhatia (Illinois Univ., Urbana). *U.S. Atomic Energy Comm.*, Rep. NP-6730, 1958, 12 pp.—Electrolytically generated $Fe^{III}$ was used as an oxidant in coulometric titrations in molten LiCl - KCl at 450° to titrate $Cr^{II}$ to $Cr^{III}$ and $V^{II}$ to $V^{III}$. The potentiometric, amperometric with one electrode, and the dead-stop methods of end-point detection are compared. Nucl. Sci. Abstr.

1143. **Improvements in gas-sampling apparatus.** G. Kent Ltd. [Inventor: G. P. Cooke]. Brit. Pat. 802,247; date appl. 23.9.55.—The gas-sampling probe comprises a sampling tube with intake and take-off sections connected to provide a liquid trap. Means are provided for heating at least that part of the sample-intake section where the outside of the tube is not subjected to direct impact of the gases being sampled to a temp. above the dew-point of the gases. J. M. Jacobs

1144. **Improvements in apparatus for detecting carbon monoxide.** Coal Industry (Patents) Ltd. [Inventors: R. Valentine and H. G. Glover]. Brit. Pat. 802,281; date appl. 6.1.56. Addn. to Brit. Pat. 756,662, dated 29.10.53.—A filter for removing carry and liquid matter from the gas being tested, and comprising filter media arranged in graded order of porosity, is arranged upstream of the differential thermometer. J. M. Jacobs

1145. **Continuous control of the carbon dioxide content of purified gases.** M. Procházka (Krásenské sklárny, Valašské Meziříčí, Czechoslovakia). *Sklář a Keramik*, 1958, **8** (6), 181–182.—A simple apparatus is described which is based on the diffusion of the gaseous mixture through a diaphragm and absorption of the $CO_2$ by an alkali hydroxide contained in a porous vessel. The difference of pressure inside and outside the vessel is related linearly to the $CO_2$ content of the sample. J. Zýka

1146. **Improvements in or relating to sonic gas analysis apparatus.** U.K. Atomic Energy Authority [Inventor: E. Duncombe]. Brit. Pat. 801,757; date appl. 22.7.55.—A sonic gas analyser tube comprises one diaphragm driven electromagnetically to excite a second diaphragm via a double cavity having its parts joined by an orifice. The diaphragms are supported in a manner allowing their free vibration, *e.g.*, on annular knife edges under magnetic forces. J. M. Jacobs

1147. **An improved liquid - liquid extractor.** P. A. Sims and G. A. Adams (Appl. Biol. Div., National Research Council, Ottawa, Ontario, Canada). *J. Amer. Oil Chem. Soc.*, 1958, **35** (3), 139–140.—An all-glass apparatus is described which is suitable for the extraction of a liquid at a series of increasing temperatures. It is provided with a water jacket, a magnetic stirrer and baffles to prevent channelling The fractionation of polymerised linseed oil with acetone in this apparatus is described. G. Burger

1148. **Portable mercury-vapour detector.** C. S. McMurray and J. W. Redmond (Union Carbide Nuclear Co., Oak Ridge, Tennessee, U.S.A.). *U.S. Atomic Energy Comm.*, Rep. Y-1188, 1958, 45 pp.—A portable, self-contained mercury-vapour detector is described and detailed diagrams are given. No external power is required, it can be operated by one person, and has replaced the commercial models in all survey work of this laboratory. Up to 0·2 mg of Hg per cu. metre of air can be detected.
K. R. C.

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*See also Abstracts*—**977**, Apparatus for fractionation of petroleum. **979**, Semi-automatic apparatus for determination of tetraethyl-lead in fuels.

### Chromatography, ion exchange, electrophoresis

1149. **Inorganic adsorption paper chromatography.** W. F. Pickering (N.S.W. Univ. of Technol., Newcastle College, Tighes Hill, Australia). *J. Chromatography*, 1958, **1** (3), 274–278.—Paper chromatograms of the nitrates of Ag, $Hg^{I}$, Pb, $Hg^{II}$, Bi, Cu, Cd, $Fe^{III}$, Ni, Co, $UO_2^{2+}$ and Al were developed by using aq. solvents of different pH. The results were interpreted in terms of a process of adsorption and displacement. A. R. Rogers

1150. **Some fundamental aspects of centrifugally accelerated paper chromatography.** H. J. McDonald, L. V. McKendell and E. W. Bermes, jun. (The Graduate School and Stritch School of Medicine, Loyola Univ., Chicago, Ill., U.S.A.). *J. Chromatography*, 1958, **1** (3), 259–265.—Apparatus for circular paper chromatography was constructed in which the paper could be rotated in its own plane at various speeds. The $R_F$ values were essentially unaffected by changes in rotational speed in the range 300 to 925 r.p.m. A low rate of addition of solvent (*e.g.*, 1·15 ml per min.) was shown to be important in developing satisfactory chromatograms. The shape of the developed chromatogram was elliptical rather than circular, with the major axis of the ellipse parallel to the machine direction of the paper. A. R. Rogers

1151. **Quantitative evaluation of circular paper chromatograms by indirect densitometry.** H. Erbring, P. Patt and N. Frey (Pharm.-chem. Lab., Dr. Madaus and Co., Köln-Merheim). *Arzneimittel-Forsch.*, 1958, **8** (5), 289–292.—The components of a mixture are separated on a circular paper chromatogram, individual zones are identified by being sprayed with a suitable reagent, and the developed segments are then photocopied and compared by a densitometric procedure (described). The method is claimed to be particularly suitable for the individual estimation of substances admixed with similar substances, *e.g.*, mixtures of amino acids or of the cardioactive glycosides of *Convallaria majalis*.
A. G. Cooper

1152. **Partition chromatography between vapour and liquid phases.** P. Etemad-Moghadame (Lab. Inst. Français Pétroles). *Chim. Anal.*, 1958, **40** (5), 149–155.—The theory of vapour-phase partition chromatography is stated, the apparatus and procedure used at the Institut Français des Pétroles are described, and those factors and conditions influencing the effectiveness of separation of components in the sample are discussed. Results are reported for the separation and quant. analysis of a mixture of (*i*) saturated alkanes, (*ii*) aliphatic

alcohols and (*iii*) 2:2-dimethylbutane and 3-methylpentane (with a column of pure eicosane on Celite). It is suggested that, when the sample consists of components containing hydroxyl groups, the stationary phase should be hydroxylic in type, *e.g.*, glycerol. Trace amounts of alkanes in the products can be removed by the use of longer columns. W. J. Baker

1153. **Detector tubes in gas chromatography.** K. Grosskopf (Dragerwerk, Moislinger Allee 53–55, Lübeck, Germany). *Erdöl u. Kohle*, 1958, **11** (5), 304–306.—In the separation of $C_3$ and $C_4$ hydrocarbons carried in N through a column of dibutyl phthalate on silica gel, the successive throughbreaks of propane, *n*- and *iso*-butane are detected by passing the outlet gas through a tube packed with silica gel impregnated with $CrO_3$, $SeO_2$ and $H_2S_2O_7$. The advance of the colour change yellow to green due to each hydrocarbon is noted; the lengths of the discoloured zones are, except for a short initial period, proportional to the quantities (ml) which may be read from calibration graphs for $C_3$ and $C_4$. Relative errors on synthetic mixtures were $< 10\%$, with repeatability $\pm 5\%$. Special detector tubes are available for each of the following gases and vapours in the concn. ranges indicated—ethanol 150 to 30,000, $NH_3$ 13 to 2600, $AsH_3$ 0·02 to 1, $C_6H_6$ 15 to 420, HCN 2 to 145, Cl 0·33 to 70, formaldehyde 2 to 40, nitrous gases 2·5 to 250, $COCl_2$ 0·25 to 17·5, $SO_2$ 4 to 155, $CS_2$ 13 to 320 and 320 to 3200, $H_2S$ 7 to 840, toluene 70 to 2700, trichloroethylene 18 to 370 p.p.m.; CO 0·001 to 0·3 and 0·3 to 4, $CO_2$ 0·5 to 5 and 1 to 20, hydrocarbons 0·0054 to 0·0675 and 0·1 to 1%; Hg 0·1 to 20 mg per cu. metre; $H_2O$ 0·1 to 80 mg per litre.
A. R. Pearson

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*See also Abstract*—**824,** Multiple spots with two competing anions.

## Optical

1154. **Improvements in or relating to double-beam spectrometers.** C. A. Parsons & Co., Ltd. [Inventor: A. E. Martin]. Brit. Pat. 795,473; date appl. 18.4.55.—In Brit. Pat. 666,270 the use of reciprocating mirrors arranged at a fixed angle to one another for switching each beam alternately on the slit of the monochromator was described. In the present modification the reciprocating mirrors are replaced by a stack of fixed (plane) mirrors, staggered alternately, so that the two sets lie in planes making a small angle with one another. The mirrors are mounted so that half the radiation in one beam path is directed by one set of mirrors on to the entrance slit of the monochromator, whilst half the radiation in the other beam is directed by the other set of mirrors on to the slit. An apertured, rotary shutter is arranged in the beam paths so that the monochromator slit receives radiations from each beam path alternately. J. M. Jacobs

1155. **Determination of the spectral-line intensities as a function of excitation time using ARL direct-reading apparatus. (Preliminary note.)** N. Kemp and Z. Eeckhaut (Univ., Ghent, Belgium). *Bull. Soc. Chim. Belg.*, 1958, **67** (5–6), 276–277.—In the procedure described, the spectral line is isolated in the focal plane of a 2-metre grating spectrograph by a second slit and falls on a photomultiplier tube, the resulting current from which charges a 1-$\mu$F capacitor whose voltage is continuously recorded potentiometrically through a d.c. amplifier with an input impedance of $\simeq 10^{12}$ ohms. This arrangement yields the desired intensity - time curve directly and is much quicker, with higher accuracy, than the "moving plate" method. Results for the complete burn of $SiO_2$ (10 mg) show that total intensity ($I$) depends on electrode-shape and -gap, but is independent of current intensity between 6 and 12 amp. The curve of log $I$ *vs.* log (wt. $SiO_2$) is linear between 2·5 and 10 mg.
W. J. Baker

1156. **Compact curved crystal X-ray spectrometer.** L. S. Birks, E. J. Brooks and G. W. Gourlay (U.S. Naval Res. Lab., Washington, D.C.). *Rev. Sci. Instrum.*, 1958, **29** (5), 425–426.—The spectrometer is provided with three sets of arms for focusing circles of 10, 20 and 40 cm radius and may be used with automatic drive or manual scanning. The drive mechanism backlash is such that $2\theta$ is less than 0·01°. G. Skirrow

1157. **Sensitive quantitative recording X-ray spectrometer.** G. E. B. Barstad and I. N. Refsdal (Norwegian Defence Res. Estab., Kjeller, Norway). *Rev. Sci. Instrum.*, 1958, **29** (5), 343–348.—Details are given of a sensitive X-ray spectrometer which can be used with micro-samples to simplify the valuation of quantitative results. The spectrometer parts are automatically positioned in accordance with the focusing geometry of the relatively large crystals of NaCl or LiF. An absolute sensitivity of $2 \times 10^{-10}$ g has been achieved for the elements Co to Zn. G. Skirrow

1158. **Photo-electric polarimeter.** Carl Zeiss Stiftung. Brit. Pat. 802,584; date appl. 23.1.56. Germany date appl. 26.1.55.—Various embodiments of a photo-electric polarimeter working on the flicker principle are described. In one embodiment the polariser is capable of rotation, the analyser is fixed, and the flicker light is produced by a device based on the Faraday effect and placed in front of the analyser. In another arrangement the polariser is also fixed and a second device, also based on the Faraday effect, is arranged behind the polariser to compensate for the optical rotation caused by the object to be measured.
J. M. Jacobs

## Thermal

1159. **Apparatus for determination of the Wiley melting-point.** R. A. Marmor and R. L. Ferm (Pillsbury Mills Inc., Minneapolis, Minn., U.S.A.). *J. Amer. Oil Chem. Soc.*, 1958, **35** (3), 109–110.—A heating bath is described which provides for the heating and stirring of six test-tubes for the determination of the melting-point of fats by the Wiley method. In each tube a thermometer is held stationary near the side while the tubes are rotated about their axes by means of meshing toothed collars which form a train of gears driven by a motor. A separate motor drives the bath stirrer.
G. Burger

1160. **Differential thermal analysis in a stream of carbon dioxide.** V. Šiške and I. Proks (Inst. Inorg. Technol., High-School of Chem. Technol., Bratislava, Czechoslovakia). *Chem. Zvesti*, 1958, **12** (4), 201–208.—The thermal decomposition of technically important carbonates has been studied with the use of an improved apparatus which enables the procedure to be carried out in a stream of $CO_2$. The curves obtained for siderite, magnesite, dolomite and marble show that the course of the dissociation and association $CaCO_3 \rightleftharpoons CaO + CO_2$ is influenced by the rate of heating and cooling.
J. Zýka

## Electrical

1161. **Test cell for use in the capacitive measurement of the moisture content of a fluent material [particularly grains or grain products].** The Quaker Oats Co. [Inventor: W. Grogg, jun.]. British Pat. 794,967; date appl. 17.12.56.—The test cell comprises an upright tubular casing, constituting one electrode of a condenser, having an insulated core extending axially into its upper end and forming the other electrode. A feed screw in the lower end of the casing feeds the material under test upwards past the core. The moisture content of the material is determined by measuring the variation in capacitive effect of the material moving continuously between the two electrodes across which a high-frequency current passes. J. M. JACOBS

1162. **Polarographic cell for routine analysis.** K. Kadič (Spolek Pro Chem. a Hutní, Ústí nad Labem, Czechoslovakia). *Chem. Průmysl*, 1958, **8** (5), 249.—A new type of arrangement for polarographic analysis consists of a polarographic cell with a separate calomel electrode. Both parts are connected with Cellophane and the inside of the cell is covered with a layer of silicone. J. ZÝKA

1163. **Improvements in and relating to polarographic cells.** Cambridge Instrument Co., Ltd. [Inventors: S. A. Bergen and W. J. Parker]. Brit. Pat. 796,183; date appl., 17.10.55.—The cell is provided with multiple nozzles through which the solution to be analysed is passed in discrete random drops into a mercury-pool cathode. In another embodiment the solution is passed upwards through the mercury after passing through a partition member which is pervious to the solution, but impervious to mercury, *e.g.*, fine-mesh nylon or polyethylene. The anode consists of a suitable metal, *e.g.*, silver, deposited on the underside of the partition. J. M. JACOBS

1164. **A drop-synchronised recording polarograph.** Yoshikazu Yasumori (Yanagimoto Co. Ltd., Kiyamachi, Nagagyo-ku, Kyoto). *Japan Analyst*, 1958, **7** (6), 354–359.—In order to obtain a smooth d.c. polarogram, Barker's principle (*Analyst*, 1952, **77**, 685; for a.c. polarography) was employed. When a high-frequency (455 kilocycles per sec.) current, which causes the recorder to operate during a short period, a certain time after a mercury drop falls, overlaps the d.c. potential, only a negligible change in $E_{\frac{1}{4}}$ value and in wave height is observed for diffusion-controlled reversible waves. The compensating d.c., which operates the balancing motor of a pen-recording polarograph, is introduced into the amplifier through a gate; this gate is controlled by the high-frequency current and permits the d.c. flow only for several tenths of a second, $\simeq 2$ sec. (>40% of the dropping interval) after the dropping of a mercury drop. K. SAITO

1165. **Polarograph with automatic corrector for the electrode potential.** S. Oka (Instrum. Div., Shimadzu Manf. Co., Kyoto, Japan). *Anal. Chem.*, 1958, **30** (10), 1635–1638.—The instrument described embodies an electronic servo-amplifier and an auxiliary potential applier, and automatically corrects the potential of the indicator electrode in accordance with the applied voltage. Any potential drops arising from internal and external resistances in the cell circuit are continuously compensated. Reproducibility and accuracy are within the limits of normal polarographic analysis.
K. A. PROCTOR

1166. **Polarographic determinations in micro-cells.** F. v. Sturm (Res. Lab., Siemens-Schuckertwerke A.-G., Erlangen, Germany). *J. Polarographic Soc.*, 1958, (2), 28–35.—A micro-cell that enables polarographic and coulometric measurements to be made on small vol. (0·05 ml) of soln. is described. Advantages and applications of the cell are discussed.
H. F. W. KIRKPATRICK

1167. **Behaviour of the Levin antimony microelectrode.** L. M. Garcia, W. C. Purdy and J. T. Stock (Dept. of Chem., Univ. of Connecticut, Storrs, U.S.A.). *Chemist Analyst*, 1958, **47** (2), 36–37.—The results of a critical study of the antimony - antimony oxide electrode (*cf.* Levin, *Chemist Analyst*, 1952, **41**, 89) in aq. systems are outlined, and it is concluded that this type of electrode combines simplicity with stability.
G. S. ROBERTS

1168. **Antimony - antimony potentiometric titration system.** J. T. Stock and W. C. Purdy (Dept. of Chem., Univ. of Connecticut, Storrs, U.S.A.). *Chemist Analyst*, 1958, **47** (2), 37–38.—The application of a pair of antimony electrodes to pH measurement and acid - base titration is described.
G. S. ROBERTS

1169. **Use of coulometry in metallurgical laboratories for the automation of analytical procedures.** M. Šícha (Vítkovické Železárny, Ostrava, Czechoslovakia). *Hutn. Listy*, 1958, **13** (6), 533–538.—The principle and various arrangements for coulometric analysis are described. The following coulometric determinations have been found advantageous for metallurgical analysis and are given in detail: C with electrolytically generated $Ba(OH)_2$; Si, P and S with NaOH; Mn, Cr, V and Ce with $Fe^{2+}$; Fe, V and Ce with $Ti^{3+}$; Ti and mixtures of Ti plus Fe with $Ce^{4+}$; Ca, Cu, Zn and Pb with EDTA; $Fe^{3+}$ with a complex of $Fe^{II}$ with EDTA (disodium salt). J. ZÝKA

1170. **Studies on continuous coulometric titration. III. Continuous coulometric titration with amperometric end-point indication.** Takeo Takahashi and Hiroshi Sakurai (Inst. of Ind. Sci., Tokyo Univ., Yayoicho, Chiba). *Japan Analyst*, 1958, **7** (5), 296–300.—The principle of coulometric titration with amperometric end-point indication is theoretically discussed with reference to the difference between this method and that involving potentiometric end-point indication. An example is given of the oxidation of $H_3AsO_3$ with electrolytically generated Br in 1·2% KBr in 3 *N* $H_2SO_4$. A linear relationship was obtained between the indicator current (indicator electrode, spiral-form platinum wire, 0·5 mm in diameter, 50 mm in length; applied potential 0·4 V) and the concn. of the excess of Br. The indicator current is obtained from the potential drop across a variable resistance (<30 kΩ), by which the sensitivity can be changed. K. SAITO

1171. **Improvements in or relating to physico-chemical apparatus.** N. V. Philips' Gloeilampenfabrieken. Brit. Pat. 791,088; date appl. 24.5.55. Date appl. U.S.A. 27.5.54.—The specimen to be analysed is irradiated with X-rays or $\gamma$-rays of such wavelengths that the specimen emits secondary radiation characteristic of its constituents. This secondary radiation is detected by a proportional counter and the frequencies of those pulses produced by the counter whose amplitudes lie within each of

a series of predetermined narrow ranges are counted. The characteristic pulse-amplitude corresponding to the max. pulse frequency is measured and compared with the corresponding pulse-amplitude obtained from a standard specimen.

J. M. JACOBS

1172. **Chemical dosimeter for X- and $\gamma$-radiation with good reproduction.** Z. Spurný (Inst. Nucl. Physics, Acad. Sci., Prague). *Jaderná Energie*, 1958, **4** (10), 294–296.—The construction of a new dosimeter for measurements of X- and $\gamma$-radiation in the range from 0 to 200 rads is described. It has been found that blue soln. of resazurin are converted into the red acid form as a result of the HCl set free during irradiation, and the method described is based on measurement of the change in pH. The influence of dose, dose rate, temp., radiation energy and the possibilities of colour indication have been studied. *Procedure*—Dilute the soln. of resazurin ($1 \cdot 10^{-3}$ *M* in 96% ethanol) with a mixture of $CCl_4$ and ethanol (4:1) and add to 4-ml glass ampoules. After irradiation, 0·6 ml of $H_2O$ is added, and the aq. layer containing resazurin and the HCl produced is transferred to a beaker and its pH measured with a suitable apparatus. J. ZÝKA

1173. **Portable apparatus for the detection of radioactive minerals.** V. I. Antonescu (Univ. "A. I. Cuza," Iaşi, Romania). *Ann. Ştiinţ. Univ. "A. I. Cuza," Iaşi*, 1957, **3** (1–2), 452.—The apparatus (illustrated) is very simple, and its weight, with auxiliary equipment, does not exceed 1·4 kg.

H. SHER

1174. **A three-stage mass spectrometer.** F. A. White, F. M. Rourke and J. C. Sheffield (General Electric Co., Knolls Atomic Power Lab., Schenectady, N.Y.). *Appl. Spectroscopy*, 1958, **12** (2), 46–52.—The spectrometer incorporates two consecutive magnetic focusing lenses, each of 20 in. radius, followed by an electrostatic analyser with a 20-in. radius of curvature. An abundance sensitivity of $10^{10}$ was measured at the mass-23·5 position. The instrument has been applied to the investigation of charge exchange, energy loss, cross-sections for atomic interactions, small-angle interactions and other difficult physical measurements, as well as for isotopic analysis. The double-focusing properties of the second magnet and the electrostatic lens permit the use of an arc source for the production of ions, which would be useful for the investigation of impurities in semi-conductors. P. T. BEALE

# ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

| | |
|---|---|
| alternating current | a.c. |
| ampere | amp. |
| Ångström unit | A |
| anhydrous | anhyd. |
| approximate, -ly | approx. |
| aqueous | aq. |
| atmospher-e, -ic. | atm. |
| boiling-point | b.p. |
| British thermal unit | B.Th.U. |
| calorie (large) | kg-cal. |
| calorie (small) | g-cal. |
| centimetre | cm |
| coefficient | coeff. |
| concentrated | conc. |
| concentration | concn. |
| constant | const. |
| corrected | (corr.) |
| crystalline | cryst. |
| crystallised | cryst. |
| cubic | cu. |
| current density | c.d. |
| cycles per second | c/s |
| density | $\rho$ |
| density, relative | $d$ or wt. per ml |
| dilute | dil. |
| direct current | d.c. |
| distilled | dist. |
| ethylenediaminetetra-acetic acid | EDTA |
| electromotive force | e.m.f. |
| equivalent | equiv. |
| gram | g |
| gram-molecule | mole |
| half-wave potential | $E_{\frac{1}{2}}$ |
| hour | hr. |
| hydrogen ion exponent | pH |
| infra-red | i.r. |
| insoluble | insol. |
| international unit | i.u. |
| kilogram | kg |
| kilovolt | kV |
| kilowatt | kW |
| liquid | liq. |
| maxim-um, -a | max. |
| melting-point | m.p. |
| microgram | $\mu$g (not $\gamma$) |
| microlitre | $\mu$l |
| micromole | $\mu$mole |
| micron | $\mu$ |
| milliampere | mA |
| milli-equivalent | milli-equiv. |
| milligram | mg |
| millilitre | ml |
| millimetre | mm |
| millimicrogram | m$\mu$g |
| millimolar | m$M$ |
| millivolt | mV |
| minute (time) | min. |
| molar (concentration) | $M$ |
| molecul-e, -ar | mol. |
| normal (concentration) | $N$ |
| optical rotation | $\alpha_\lambda^t$ |
| ounce | oz |
| parts per million | p.p.m |
| per cent. | % |
| per cent. (vol. in vol.) | % (v/v) |
| per cent. (wt. in vol.) | % (w/v) |
| per cent. (wt. in wt.) | % (w/w) |
| potential difference | p.d. |
| precipitate (as a noun) | ppt. |
| precipitated | pptd |
| precipitating | pptg. |
| precipitation | pptn. |
| preparation | prep |
| qualitative, -ly | qual. |
| quantitative, -ly | quant. |
| recrystallised | recryst. |
| refractive index | $n_\lambda^t$ |
| relative band speed | $R_F$ |
| relative humidity | r.h |
| revolutions per minute | r.p.m. |
| saponification value | sap. val. |
| saturated calomel electrode | S.C.E. |
| second (time) | sec. |
| soluble | sol. |
| solution | soln. |
| specific gravity | sp. gr. |
| specific rotation | $[\alpha]_\lambda^t$ |
| square centimetre | sq. cm |
| standard temp. and pressure | s.t.p. |
| temperature | temp. |
| ultra-violet | u.v. |
| vapour density | v.d. |
| vapour pressure | v.p. |
| volt | V |
| volume | vol. |
| watt | W |
| wavelength | $\lambda$ |
| weight | wt. |

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

| | | | |
|---|---|---|---|
| greater than | $>$ | less than | $<$ |
| not greater than | $\ngtr$ | not less than | $\nless$ |
| is proportional to | $\propto$ | of the order of, approximately | $\simeq$ |

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, $Fe^{II}$, $Mo^{V}$. Substances in the ionic state are represented by $Na^+$, $Fe^{2+}$, $Fe^{3+}$, etc., for cations and by $Cl^-$, $SO_4^{2-}$, $PO_4^{3-}$, etc., for anions.

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## CONTENTS

| | Abstract |
|---|---|
| **General Analytical Chemistry** | |
| Reviews; reagents; methods of general application | 810 |
| **Inorganic Analysis** | |
| General methods; elements (in order of the Periodic Table); minerals; industrial products | 824 |
| **Organic Analysis** | |
| Elements and radicals; organic compounds; industrial products | 952 |
| **Biochemistry** | |
| BIOLOGICAL FLUIDS; ANIMAL AND VEGETABLE TISSUES | 994 |
| PHARMACEUTICAL ANALYSIS | 1065 |
| FOOD | |
| Food additives; beverages; edible oils and fats; vitamins | 1093 |
| SANITATION | |
| Air; water; sewage; industrial wastes | 1124 |
| AGRICULTURAL ANALYSIS | |
| Soil; fertilisers; herbicides; pesticides; animal feeding-stuffs | 1132 |
| **General Technique and Apparatus** | |
| GENERAL | 1141 |
| CHROMATOGRAPHY; ION EXCHANGE; ELECTROPHORESIS | 1149 |
| OPTICAL | 1154 |
| THERMAL | 1159 |
| ELECTRICAL | 1161 |

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